

1993

Lipid oxidation in dry corn masa flour and strategies to improve shelf-life

Reyna Luz Vidal-Quintanar
Iowa State University

Follow this and additional works at: <https://lib.dr.iastate.edu/rtd>

 Part of the [Agriculture Commons](#), and the [Food Science Commons](#)

Recommended Citation

Vidal-Quintanar, Reyna Luz, "Lipid oxidation in dry corn masa flour and strategies to improve shelf-life " (1993). *Retrospective Theses and Dissertations*. 10561.
<https://lib.dr.iastate.edu/rtd/10561>

This Dissertation is brought to you for free and open access by the Iowa State University Capstones, Theses and Dissertations at Iowa State University Digital Repository. It has been accepted for inclusion in Retrospective Theses and Dissertations by an authorized administrator of Iowa State University Digital Repository. For more information, please contact digirep@iastate.edu.

9 4

1 4 0 3 0

U·M·I
MICROFILMED 1994

INFORMATION TO USERS

This manuscript has been reproduced from the microfilm master. UMI films the text directly from the original or copy submitted. Thus, some thesis and dissertation copies are in typewriter face, while others may be from any type of computer printer.

The quality of this reproduction is dependent upon the quality of the copy submitted. Broken or indistinct print, colored or poor quality illustrations and photographs, print bleedthrough, substandard margins, and improper alignment can adversely affect reproduction.

In the unlikely event that the author did not send UMI a complete manuscript and there are missing pages, these will be noted. Also, if unauthorized copyright material had to be removed, a note will indicate the deletion.

Oversize materials (e.g., maps, drawings, charts) are reproduced by sectioning the original, beginning at the upper left-hand corner and continuing from left to right in equal sections with small overlaps. Each original is also photographed in one exposure and is included in reduced form at the back of the book.

Photographs included in the original manuscript have been reproduced xerographically in this copy. Higher quality 6" x 9" black and white photographic prints are available for any photographs or illustrations appearing in this copy for an additional charge. Contact UMI directly to order.

U·M·I

University Microfilms International
A Bell & Howell Information Company
300 North Zeeb Road, Ann Arbor, MI 48106-1346 USA
313/761-4700 800/521-0600

Order Number 9414030

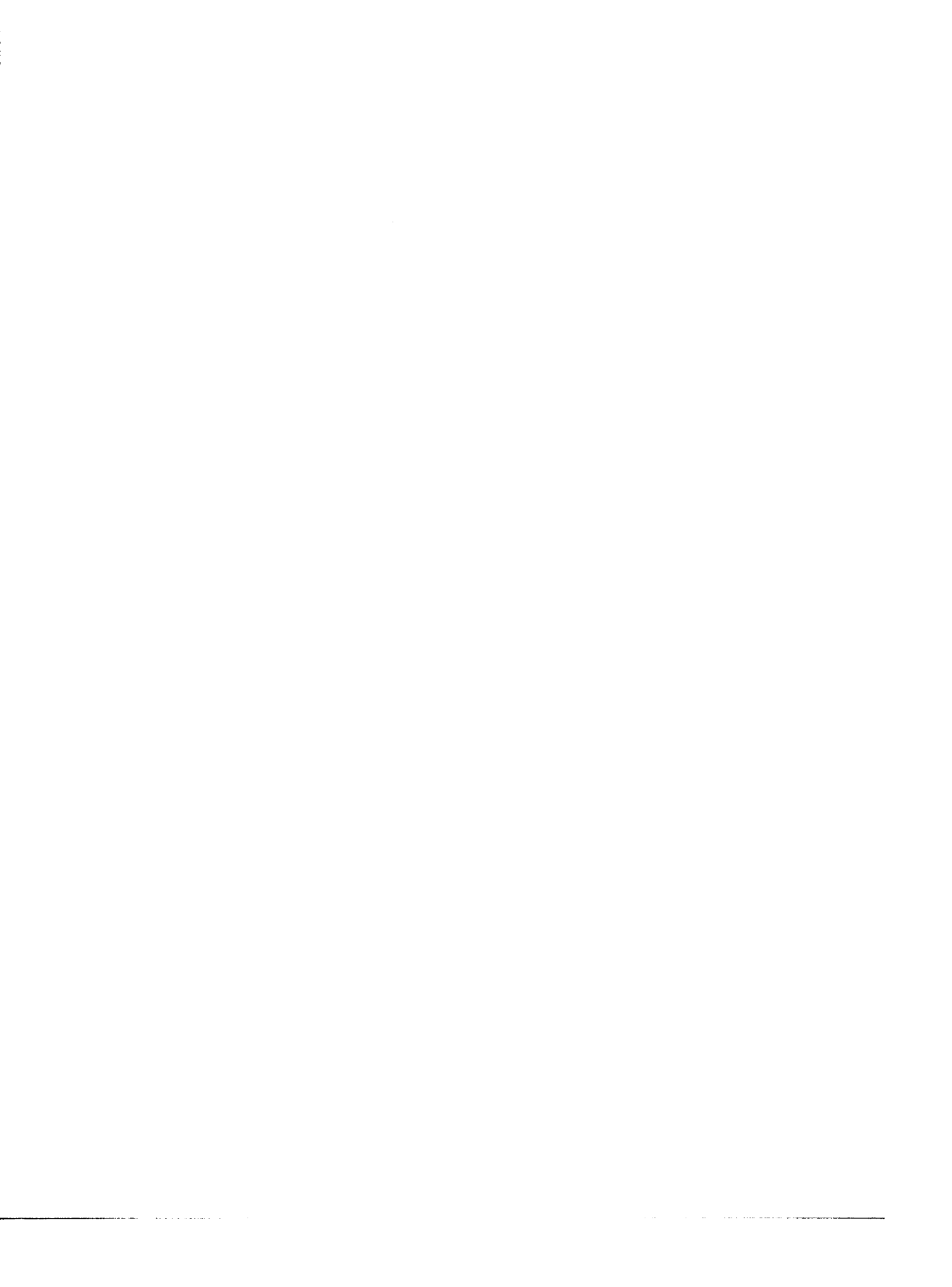
Lipid oxidation in dry corn masa flour and strategies to improve shelf-life

Vidal-Quintanar, Reyna Luz, Ph.D.

Iowa State University, 1993

Copyright ©1993 by Vidal-Quintanar, Reyna Luz. All rights reserved.

U·M·I
300 N. Zeeb Rd.
Ann Arbor, MI 48106



**Lipid oxidation in dry corn masa flour and strategies
to improve shelf-life**

by

Reyna Luz Vidal-Quintanar

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of the
Requirements for the Degree of
DOCTOR OF PHILOSOPHY

**Department: Food Science and Human Nutrition
Major: Food Science and Technology**

Approved

Signature was redacted for privacy.

In Charge of Major Work

Signature was redacted for privacy.

For the Major Department

Signature was redacted for privacy.

For the Graduate College

Members of the Committee:

Signature was redacted for privacy.

**Iowa State University
Ames, Iowa
1993**

Copyright © Reyna Luz Vidal-Quintanar, 1993. All rights reserved.

TABLE OF CONTENTS

GENERAL INTRODUCTION	1
----------------------	---

PAPER I. LIPID OXIDATION OF DRY CORN MASA FLOUR

ABSTRACT	17
INTRODUCTION	18
MATERIAL AND METHODS	20
RESULTS AND DISCUSSION	24
CONCLUSIONS	32
LITERATURE CITED	33

**PAPER II. ROLE OF OIL ON PHYSICAL PROPERTIES
OF DRY CORN MASA FLOUR AND SENSORY
CHARACTERISTICS OF CORN TORTILLAS**

ABSTRACT	61
INTRODUCTION	62
MATERIAL AND METHODS	64
RESULTS AND DISCUSSION	69
CONCLUSIONS	73
LITERATURE CITED	74

**PAPER III. ROLE OF CORN GERMS ON PHYSICAL PROPERTIES
OF DRY CORN MASA FLOUR AND SENSORY
CHARACTERISTICS OF CORN TORTILLAS**

ABSTRACT	88
INTRODUCTION	89
MATERIAL AND METHODS	91
RESULTS AND DISCUSSION	94
CONCLUSIONS	97
LITERATURE CITED	98
GENERAL SUMMARY	106
LITERATURE CITED	108
ACKNOWLEDGEMENTS	114
APPENDIX	115

GENERAL INTRODUCTION

Literature Review

Archaeological evidence indicates that corn existed as a wild precursor of cultivated maize some 7,000 years ago in central Mexico (Bressani 1990). Approximately 2,000 years later, corn was under cultivation in this region. Corn domestication, cultivation and transformation into edible products have been important to Mexican cultures and the central subject of paintings, poetry, songs and religious ceremonies.

Lime cooking of corn is a well-known process to make a variety of corn products such as tortillas, tacos, nachos, tostadas and other corn snacks (Bedolla and Rooney 1982). The tortilla-making process from dry corn masa flour is receiving increased attention because commercial tortilla plants face the difficult task of converting the traditional home-level hand-made nixtamal method into a large-scale industrial process.

Dry corn masa flour has longer shelf-life than wet masa as well as greater convenience in home and commercial preparation. The dry corn masa flour industry quickly overcame early problems such as lack of flavor and poor texture. Many researchers have made significantly contributions to understanding the effects of lime cooking on corn components (Gomez et al 1987); component

functionality (Bressani et al 1958, Robles et al 1988), and to making processing improvements (Bedolla and Rooney 1984).

Nixtamalization

The traditional lime-cooking method originated in México (Katz et al 1974), and expanded to the United States during the last decade. Today, even at the industrial level, the three basic steps of cooking, soaking and grinding rely on empirical parameters and/or experienced personnel (Bedolla and Rooney 1982).

The three main steps for preparing corn masa and tortilla making have remained largely unchanged. Variations in cooking time, amount of water, and lime concentration have been discussed by Bressani 1990. Traditional alkali-cooked corn products are prepared by cooking (95°C/60-100 min) whole corn kernels in a calcium hydroxide solution (0.58-3.00% corn weight base) and steeping the nixtamal (23-32°C/12 to 18 hr). The resulting nixtamal is washed to remove pericarp, then ground with stone or hammermills. The dough is shaped into thin disks and cooked into tortillas. This operation can be done by hand or with industrial machinery.

Industrial Processing

Migration of people from rural to urban areas increased the demand for ready-made tortillas and for precooked flours to be made into tortillas (Bressani

1990). These growing demands led to the development of equipment to process raw corn into cooked masa and industrial production of corn tortillas. The first rotary mill and tortilla maker were designed by Romero in 1908. This equipment was later replaced by a more efficient model, the Celorio Sheeter (Anonymous 1979). In the basic process, the dough is passed through a series of rotating metal rolls which cut the flattened dough into circular disks. The shaped dough falls onto a moving belt or continuous cooking griddle, and then the tortillas are deposited into a receptacle at the end of the belt.

A industrial tortilla-making processing involves using either wet corn masa, cooked as previously indicated, or rehydrating dry corn masa flour. The industrial process has been described by various authors (Paredes and Saharopulos 1983, Gomez et al 1987). The manufacturing of dry corn masa requires two operations of high energy input; cooking the corn and drying the cooked corn dough. The ground masa is dried using tower air suspension or belt drying methods (Molina et al 1977). Hammermilling and particle size classification are the final steps (Gomez et al 1989). Table tortillas require finely ground flour in order to properly puff during baking. Fried tortillas require a larger mean particle size than table tortillas to avoid blister formation, high oil uptake, and excessive breakage during packaging and handling (Bedolla and Rooney 1982).

The physical and chemical specifications of dry corn masa flour set by the Mexican government include moisture (10% maximum), ash (2% db maximum),

protein (7.5% db minimum), lignin (negative reaction) a folding test (no cracks). In addition, 85% of the flour must pass through a 60-mesh screen (250 μm) (Bressani 1990).

Technological Modification

Any method which reduces preparation time and cost, and still yields acceptable tortillas, would be highly desirable (Bressani 1990). Efforts toward this end have been made by numerous researchers. Bedolla et al (1983) tested various methods of cooking corn and showed that the cooking method affected total dry matter loss. Morad et al (1986) concluded that 40% reduction in cooking time could be achieved by presoaking the grain before alkali cooking. Presoaking significantly increased dry matter loss, water uptake, calcium content, and enzyme-susceptible starch, and decreased viscosity of the masa slurry.

Khan et al (1982) reported on a pressure cooking method which produced sticky, over-gelatinized, and dark-colored masa. Sticky masa is largely due to over-gelatinized starch and is difficult for equipment to handle. The same study showed that tortillas made from over-cooked nixtamal had an undesirable rubbery texture. Tortillas from undercooked nixtamal were more crumbly than those from optimally-cooked nixtamal.

Johnson et al (1980) tested a micronization process to produce dry corn masa flours. This study showed that micronized-corn tortillas had textures and

rollabilities comparable with tortillas made from commercial corn dry masa flour and from fresh wet corn masa. Micronized dry corn flour showed greater water uptake and high enzyme-susceptible starch compared with commercial dry corn masa flour. Micronization reduced mold growth and caused rancidity (ketonic rancidity) of the flours.

Extrusion cooking has also been evaluated as alternative technology to produce nixtamalized corn masa (Basua et al 1979). Extrusion cooking gave good dough properties. Organoleptic properties and chemical compositions of tortillas made from extruded masas were comparable with those made by the traditional process.

Effect of Lime-Cooking Treatments on Corn Components

During lime-cooking, corn kernels are subjected to high temperatures and alkaline solution. Nutrient losses are caused by leaching when corn is soaked or cooked in water. High temperatures of cooking and alkalinity accelerate nutrient losses (Bressani 1990).

Carbohydrates. Raw corn and corn tortillas contain significant amounts of soluble carbohydrates (Pflugfelder et al 1988). About 5% of the starch is lost during the transformation of corn into corn tortillas. Robles et al (1988) found that alkali-cooking and soaking corn increased viscosity in the flour slurry. Cooking time

significantly affected pasting properties although there was no extensive gelatinization of the starch. The same authors reported similar gelatinization endothermies for raw corn as for nixtamal flours. They suggested that starch is stabilized by calcium and amylose interaction, and therefore, gelatinization was restricted (Robles et al 1986). Morad et al (1986) found that enzyme-susceptible starch increased as cooking time increased.

Protein. Lime treatment and cooking of tortillas did not greatly affect total crude protein or total amino acid content of corn masa flours (Sandeson et al 1978). Vivas et al (1990) found that cooking, steeping, grinding and baking of tortillas decreased in-vitro protein digestibilities (using pepsin) of corn. But, only small changes have been reported in essential amino acids (Ortega et al 1986, Bressani and Scrimshaw 1958). Cross-linking of prolamins decreased protein digestibility. Protein digestibility of tortillas improved when reducing agents, that break inter- and intra-molecular disulfide bonds, were utilized (Vivas et al 1990). Other studies have shown that nixtamalization decreased the amount of protein extraction compared with raw corn (Vivas et al 1987).

Nixtamalization and baking of the masa during tortilla making induced hydrophobic interactions, peptide cross-linking, protein complexing, and denaturation of globulins, zeins, and glutelin-like polypeptides (Ortega 1986, Paredes and Saharopulus 1982). The complexes formed probably made the

protein less soluble and less available for enzyme action, as well as affecting the molecular weight distribution of the protein complex in nixtamalized corn as compared with raw corn proteins (Vivas et al 1987). Katz et al 1974 showed that alkali cooking of corn resulted in higher glutelin solubility, higher lysine and niacin contents, and improved the ratio of isoleucine to leucine. Ortega et al (1986) found that available lysine was reduced during tortilla making.

Lysino-Alanine Formation (LAL). Formation of new peptides by cross-linking reactions, such as lysino-alanine, lanthionine and ornithoalanine lowers protein quality and biological availability (Mauron 1977). LAL causes nephrocytomegalia in rats, but no renal changes have been encountered in other animals (dogs, monkeys) (DeGroot et al 1976).

Chu et al (1976) found very little LAL formation when corn was cooked in calcium solution, as compared with sodium or potassium salts. Calcium ions interfere with LAL formation by binding to certain amino acid sequences or by blocking certain functional groups in the side chains of proteins. Sanderson et al (1978) reported lanthionine and ornithine formation during alkali cooking of corn. These authors found no LAL in common white corn or in high-lysine corn; however, 0.05 and 0.49% LAL of the total protein were found in the respective alkali-treated corn products. However, since tortillas have been eaten for a long time, it would

seem that the small amounts of LAL present do not drastically affect the nutritive value or cause any pathological effects (Bressani 1990).

Lipids. Pflugfelder et al (1988b) reported that 25-50% total masa lipid was free and partially emulsified. The same authors found losses of 11.8 to 18.1% in the oil content during the processing of maize to masa. Presumably, some of the oil becomes bound to protein or starch. Corn oil contains about 24% oleic and 62% linoleic acid (Hamilton 1989). Corn oil is regarded as being high in quality for cooking oil, margarine, salad oil, shortenings, mayonnaise, frying potato chips, and use in sauces and soups. The fatty acid compositions of several cultivars did not change much from raw corn to tortillas (Bressani 1990).

Lipoxygenases from the germ tissue can oxidize the unsaturated fatty acid linoleic acid (Inglet 1970). Longer storage leads to oxidative rancidity that is caused by autoxidation of unsaturated fatty acids. Oxidation of unsaturated lipids involves a free radical reaction with an initiation step, a propagation step and a termination step. Lipid oxidation involves formation of peroxy radicals and hydroperoxides which undergo chain scission to more stable compounds, such as aldehydes, hydroxy-acids, and/or epoxides (Eriksson 1987, and Pokorny 1987). Products of chain scission are known to produce off flavors and off odors in food. Schaich and Karel (1974) reported that some free radicals were transferred from oxidizing methyl linoleate to proteins (cystine, arginine, histidine, tryptophan and

lysine amino acids). Oxidized lipids reacted with proteins to form brown-colored macromolecular products (Eriksson 1987).

The type of fatty acids combination in the triglyceride and the type of fatty acids in the 1, 3 sn-position of the triglyceride affect the rate of lipid oxidation and nutritional value of the final product (Weber 1978). The fatty acid in the outer (sn 1 and 3) positions of the triglyceride is more susceptible to oxidation than the sn-2 position. Animals easily incorporate in their lipids the fatty acid from the sn-2 of dietary fat. Enzyme cleavage is faster on the fatty acid from the outer position resulting in an easy absorption of the 2-monoglyceride (Weber 1978). If breeding could concentrate unsaturated fatty acids in the sn-2 position, lipid oxidation could be reduced and feed efficiency improved (Weber 1978).

Crude Fiber. Alkali cooking reduces insoluble dietary fiber content because pericarp tissue is removed during cooking and washing. The cooking process increases the amount of soluble polysaccharides (gums, pectins and some hemicellulose) by disrupting cell walls (Serna-Saldivar 1987).

Vitamins. Bressani et al (1958) reported losses of thiamine, riboflavine, niacin, and carotene during preparation of tortillas as compared with raw corn. Niacin has attracted the most attention, because of its relationship to pellagra (Bressani 1990).

Alkaline treatment releases covalently bound niacin from sugars, peptides and phenolic compounds (Bressani et al 1958).

Bressani et al (1961) reported that after lime-cooking of corn the endosperm tissue contributes 68% of total niacin while 5.5% comes from the germ, and 26% is eliminated in the nejayote. Furthermore, they reported that in-vitro enzymatic hydrolysis with pepsin-pancreatin digestion liberated all the niacin from both raw corn and from tortilla. They concluded that the amino acid balance rather than bound niacin was responsible for the difference between raw and lime-processed corn in biological active and pellagragenic action. The same authors found that tortillas generally had a better growth promoting value in rats than raw corn.

Minerals. McDonough et al (1987) reported than more calcium was absorbed in the pericarp than other parts of the kernel. They concluded that a relationship existed between calcium content and cell wall degradation. Calcium plays an important role in tortilla making. First, calcium maintains the pH of the cooking water mixture at about 12.4. Alkaline pH is necessary to hydrolyze the hemicellulose of the pericarp. The amount of hemicellulose is indirectly measured by a pericarp removal test (Serna-Saldivar et al 1990). Second, the alkali medium favors changes in protein conformation (Bressani and Scrimshaw 1958), starch gelatinization (Trejo-Gonzales et al 1982, Paredes-Lopez and Saharopulos 1982) and release of bound niacin to a more biologically available form (Harper et al

1958). Finally, the absorbed calcium also contributes to the flavor of the products made from alkali-cooked corn (Morad et al 1986).

The calcium content of the lime-cooked corn is 0.14 to 0.15 g/kg (Bressani 1958, Serna-Saldivar 1987). Corn tortillas have significantly more calcium than raw corn (Bressani 1958, Bedolla and Rooney 1982, Pflugfelder et al 1988). Serna-Saldivar (1987) reported 28.3-47.2% calcium retention in nixtamal after alkaline cooking of the corn. Recent studies with rats have shown that calcium of corn tortillas is highly available to growing rats. The level of dietary calcium from tortillas greatly affects the calcium and phosphorus absorption rate (Serna-Saldivar et al 1991).

Physicochemical Characteristics of Alkali-Cooking of Corn

Grain Selection. There is little knowledge about the physical/chemical properties of corn that can be used for hybrid screening and/or quality control. The selection and price of corn for alkali-cooking are affected by percentage of defective grain, moisture content, and percentage of impurities, such as dirt, cobs, leaves, etc (Bedolla and Rooney 1982, Jackson et al 1988, Bressani 1990). Paredes and Saharopulos (1982) reported that soundness (<4% cracked kernels, not presence of insect damages, or mold growth) of the kernel is one of the most important factors in the production of tortillas with optimum organoleptic (flavor and texture) and functional characteristics (not sticky dough). Bedolla and Rooney (1982)

reported that corn with more than 4% cracked kernels produces sticky masa. In addition, these authors found that horny endosperm had higher optimum water uptake and starch gelatinization than floury endosperm.

Jackson et al (1988) reported that broken kernels increase dry matter loss and chemical oxygen demand of the nejayote (cooking water). Losses of starch, protein, and lipids were greater when damaged corn and softer corn hybrids were processed (Pflugfelder et al 1988b). Bedolla and Rooney (1983) found that the texture of the dough was affected by endosperm texture, type, drying method, storage, and soundness of the corn kernel. Martinez-Herrera and Lachance (1979) established a functional relationship between kernel hardness and time needed for optimum cooking.

Dry Matter Loss (DML). DML resulting from alkaline-cooking of corn constitutes a good index of corn quality for tortilla making (Jackson et al 1988). The DML during nixtamalization comes from lime-treatment, heating, and leaching of corn solids. Jackson et al (1988) reported that greater losses were obtained from stress-cracked and broken kernels than from sound kernels. They concluded that high steeping temperatures and high lime levels increased dissolved solids in wastewater.

Gomez et al (1989) reported that the most evident effects of alkaline cooking on gross structure of the kernel were loosening and removal of the

pericarp. The pericarp being peeled away was found to be one-third of the total DML. The loss of solids when converting from raw corn to nixtamal varied from 5-14% (Bressani et al 1958, Khan et al 1982, Morad et al 1986, Jackson et al 1988). Bressani et al (1958) found that DML included about one-third of the ether-extractable portion of corn, one-fourth of the crude fiber content, one-tenth of the nitrogen content and one-fifth to one-half of the vitamins and minerals.

Water Uptake and Starch Gelatinization. Morad et al (1986) reported that after soaking in lime solution (1%) at 25°C for 12 hr, corn kernels absorbed about 45% of their original weight. While alkali-cooking at 92-95°C with the same level of lime (1%) and water for 100 min, the corn kernels absorbed approximately 90% of their original weight in water. Higher water uptake of corn during nixtamalization promotes chemical changes in starch and favors dough formation.

Gomez et al 1989 showed that nixtamalized corn kernels exhibited swelling and hydration of the starch granules and protein. The starch absorbed water and swelled, filling the inner area within each endosperm cell. Cooking furnished the energy needed to break intramolecular hydrogen bonds of the starch granules. The starch granule will begin to swell rapidly with progressive hydration, and lose of birefringence. This process is known as gelatinization (Whistler and Daniel 1985). Continuous heating causes loosening of the carbohydrate meshwork, allowing additional water to enter and enlarge the granule further (Smith 1982).

The reduction of crystalline starch granules was greatest during transition from masa to tortilla (30-40%), followed by nixtamalization (15-26%) and milling (1-10%) (Gomez et al 1989). They also reported that the loss of birefringence of starch granules was up to 15% during tortilla baking. Khan et al (1982) and Morad et al (1986) reported that the enzyme-susceptible starch value was the best index to characterize the extent of starch gelatinization in dry corn masa flours.

Objectives

The objectives of this dissertation are to determine the extent of lipid oxidation in dry corn masa as sold in the marketplace; to determine the shelf-life of dry corn masa with respect to lipid oxidation; and to determine the effect of corn oil and germ proteins on physical properties and sensory characteristics of corn tortillas.

An Explanation of the Dissertation Organization

This dissertation consists of three papers for submission, written in format required by the journal Cereal Chemistry. There is a General Summary following the three papers. The references cited in the General Introduction follow the General Summary.

PAPER I

LIPID OXIDATION OF DRY CORN MASA FLOUR

Lipid Oxidation of Dry Corn Masa Flour¹

Reyna Luz Vidal-Quintanar², Lawrence A. Johnson^{2,3,4},

Mark H. Love³, and Jane A. Love³

¹ Journal Paper J- of the Iowa Agriculture and Home Economics Experiment Station, Ames, IA, 50011. Project 0178. Research supported by the Center for Crops Utilization Research and the Iowa Agriculture and Home Economics Experiment Station.

² Graduate Research Assistant and Professor-in-Charge, respectively, Center for Crops Utilization Research, Iowa State University, Ames, IA, 50011.

³Professor, Associate Professor and Associate Professor, respectively, Department of Food Science and Human Nutrition, Iowa State University, Ames, IA, 50011.

⁴ To whom correspondence should be addressed.

ABSTRACT

A retail marketplace survey showed that the oil of dry corn masa flour is typically quite oxidized (PV from 14.2 to 65.9 meq/kg). Rancidity of dry corn masa flours was greatest during summer and least during winter. The shelf-lives of two Mexican dry corn masa flours were studied, in which these flours were stored at 15, 25, 35, 45, and 55°C in two 23-wk storage periods. Lipid oxidation was evaluated by using peroxide value (PV), conjugated dienoic acids (CD), and sensory analyses of both flours and tortillas. Increased storage time significantly increased lipid oxidation ($P \leq 1\%$), and increased storage temperature significantly decreased shelf-life (sensory score ≥ 3) at $P \leq 1\%$. Shelf-life of dry corn masa flour was 5.8 mo when stored at 25°C. A 10°C increase in temperature reduced the shelf-life of the flour by about one-half. Generally, CD and PV were poorly correlated with sensory scores of flours and tortillas. Thus, PV and CD were not good predictors of sensory detection of rancidity and consumer acceptability of dry corn masa flour.

INTRODUCTION

The populations of many Latin American countries consume relatively large amounts of corn that is processed by lime cooking and made into tortillas (Bressani 1990). Tortillas are an important staple food and contribute immensely to the diets of these consumers. Masa for tortillas is traditionally made by cooking corn in calcium hydroxide solution to produce nixtamal. Nixtamal, which has been soaked overnight, is stone ground to masa and is flattened into thin disks or tortillas for baking. The traditional process requires about 24 hr and considerable labor, and the product produced must be consumed within 1-2 days.

Recently, advanced processing technologies to produce instant dry masa flours have been developed. Such processing systems involve lime cooking, milling, drying and sifting. Dry corn masa flour has much longer shelf-life than traditional wet corn masa, and considerably less work is involved in consumer preparation (Gomez et al 1987). Recent migration of people from rural to urban areas of Latin America has greatly increased the demand for ready-made tortillas and for precooked flours to be made into tortillas.

Typical dry masa flour has 10-12% moisture and is stable against microbial growth (Carrillo-Perez et al 1990); however, it is highly susceptible to the development of off-flavors and odors usually associated with lipid oxidation of unsaturated fatty acids. Major factors that affect lipid oxidation of foods include the presence of catalysts, light, oxygen concentration, water activity, and temperature.

Exposure to high temperature during storage and distribution would likely increase the reaction rate. Therefore, the storage temperature could be managed to extend the shelf-life of dry corn masa. In a 1972 consumer and retailer survey in Mexico, unacceptable flavors and odors were reportedly present in dry corn masa flours after about 3 mo of storage during summer (Bressani et al 1990).

It is also well documented that secondary products, as well as peroxides and lipid free radicals, react with vitamins and proteins causing losses in nutritional value and in solubility (Fennema 1989, Pokorny 1987). Peroxides can react with other food constituents, or they may decompose to volatile secondary products such as aldehydes, esters, and low molecular weight aliphatic acids and ketones, responsible for off-flavors and odors.

The objectives of present work were to determine the extent of lipid oxidation in dry corn masa as sold in the retail marketplace and to determine the shelf-life of dry corn masa with respect to lipid oxidation.

MATERIALS AND METHODS

Lipid Oxidation in Dry Corn Masa Found in the Retail Marketplace

Five brands of dry corn masa flours were purchased from the Mexican and US retail markets. Maseca (mas) (Harinera de Maiz S.A. de C. V. Cd. Obregon, SON, México) and Minsa (min) (Maiz Industrializado CONASUPO, Los Mochis SIN, México) were purchased in Sonora, México. Maseca (Azteca Milling Co., Edinburg, TX) purchased in Arizona (maa), Texas (mat), and Iowa (mai); Quaker (Quaker Oats Co., Chicago, IL) purchased in Arizona (qua) and Iowa (qui); and Pioneer (Pioneer Flour Milling, San Antonio, TX) purchased in Texas (pio) were from the US market. Samples of each brand were collected every 3 mo for a year.

Storage Study

Samples. Dry corn masa flours were donated by Harinera de Maiz (Cd. Obregon, SON, México) and Minsa (Maiz Industrializado CONASUPO, Los Mochis, SIN México). The flours were sampled directly from the production lines of the two Mexican plants on the same day.

Storage Conditions. Ninety kg (1-kg packages) of each flour was mixed in a ribbon mixer (Marion Mixer model B224-1, Rapid Machinery Co., Marion, IA), and 150 ± 10 g of flour were placed in each of 530 Mason jars for each flour (410 for chemical analysis, 120 for sensory analysis). The jars were then placed in

temperature-controlled incubators at 15, 25, 35, 45, and 55°C. Samples were stored for a total storage period of 23 wk. One-half of the sample was stored at -24°C and the experiment was replicated 5 mo later. Insufficient incubator space and the large number of analyses prevented simultaneous replication. Samples (an entire jar) were taken at each temperature every 4 days for chemical analyses, for a total of 41 times, and every 2 wk for sensory analysis, for a total of 12 times.

Sensory Analyses. A trained panel consisting of five male and seven female adults (half American and half international) evaluated the flours and tortillas for rancid aromas and flavors. The panel was selected from Iowa State University students and staff. Training sessions were 20 min each using fresh (PV=14 meq/kg), lightly oxidized (PV=40-50 meq/kg), and very oxidized (PV>100 meq/kg) samples of dry corn masa flour. Panelists were served freshly cooked tortillas to be evaluated using a 5-point category scale, where 5=extremely rancid, 4=very rancid, 3=rancid, 2=slightly rancid, and 1=not rancidity (Appendix A1) (Moskowitz 1980, Stone and Sidel 1985). Training was conducted until individual scores did not vary more than 0.25 units from the mean score.

The flours and tortillas were evaluated in separate panels. A set of five flour or five tortilla samples (one from each temperature treatment) were presented in random order under red light to the panelists. Panelists evaluated each treatment by placing a mark on a 5-point category scale (Appendix A1), which best described

the rancid aroma or flavor of the sample. Testing was done in an air-conditioned panel room equipped with individual booths. Each panelist was provided with water to rinse the palate prior to tasting each sample and a cup to expectorate.

Flours. Flours (8 ± 0.5 g) were weighed into vials (23 x 85 mm) and warmed to 45°C for 30 min. A set of five vials of flours, one for each storage temperature, was presented to each panelist. Panelists smelled the flours and rated them on a 5-point category scale, where 5=extremely rancid, 4=very rancid, 3=rancid, 2=slightly rancid, and 1=not rancidity.

Tortillas. Dry corn masa flour (150 ± 5 g) and 1.15% w/w water were mixed in a kitchen mixer (KitchenAid, Inc, St. Joseph, MI) equipped with a flat beater for 30 sec at low speed and 30 sec at high speed. The dough was divided into 7.0 ± 0.25 g lots and kept in Ziploc® polyethylene bags. Doughs were pressed in a hand-press (Corona 1-F, Industrias Monterrey, Monterrey, NL, Mexico). Shaped tortillas (7.0 ± 0.25 cm in diameter) were cooked on both sides for about 2.5 ± 0.10 min using a hot iron plate.

Three tortillas per set were wrapped in a napkin and then in aluminum foil to prevent drying. A group of five sets of tortillas (one group from each storage temperature) was presented to each panelist on a hot tray to maintain serving temperature of 45°C (Salton model 3006, Pifco Limited, Failsworth, Manchester,

England) under red lights. Panelists evaluated only the middle tortilla. A 5-point category scale was used (Appendix A2), where 5=extremely rancid, 4=very rancid, 3=rancid, 2=slightly rancid, and 1=no rancidity.

Chemical Analyses

Peroxide Value (PV) and Conjugated Dienoic Acids (CD). Oil was extracted from dry corn masa flour with hexane for 5 hr using a Soxhlet extractor (Precision Instrument Co., Chicago, IL). The solvent was evaporated in a rotary evaporator under water aspiration vacuum. Lipid oxidation of the oil was assessed using AOCS Method cd8-53 for PV and Method Ti 1a-64 for CD (AOCS 1985).

Experimental Design

A 5 x 2 randomized complete block design, Temperature (15, 25, 35, 45, and 55°C) x Flour (Maseca and Minsa), was used with the 23-wk storage period defined as a block treatment. Analysis of variance (ANOVA) and correlation analysis were used to evaluate treatment effects (SAS Institute, Inc. 1989). Flours and temperature groupings in each storage period and sample codes were randomly organized.

RESULTS AND DISCUSSION

Lipid Oxidation in Dry Corn Masa Found in the Retail Marketplace

Dry corn masa flour, sampled from the processing line and immediately analyzed, had low levels of lipid oxidation, PV <15 meq/kg (Table I). However, the extent of lipid oxidation found in flours obtained from the retail marketplace was highly variable, with many being highly oxidized and few having little oxidation (Table II). Maseca from Texas (PV=14.2 meq/kg), Quaker from Iowa (PV=18.3 meq/kg), and Quaker from Arizona (PV=22.8 meq/kg) had consistently low PVs during the one-year survey. Pioneer from Texas (PV=63.5 meq/kg) and Maseca from Iowa (PV=65.9 meq/kg) were the most oxidized flours, having consistently high PVs during the one-year survey. The package size and packaging material were similar for all brands (1-2.5 kg in double paper bags). Thus, the observed differences were not attributed to packaging. More likely, differences in the storage temperature during distribution and turnover rates were important causative factors.

The season of the year significantly ($P < 1\%$) affected the PVs of samples purchased from both countries. PVs ranged 5-70 meq/kg during winter and 10-99 meq/kg during summer (Table II). In general, flours purchased during spring (PV=50.2 meq/kg) and summer (PV=45.8 meq/kg) showed the highest lipid oxidation level; and the least amount of oxidation was measured in samples purchased during winter (PV=32.4 meq/kg).

Flours purchased in México were slightly more oxidized than those purchased in the United States (mean values PV=46.4 vs 38.8 meq/kg, respectively). It was surprising that the variation in lipid oxidation was so great within the same location. For instance, Quaker had much less oxidation than Maseca even though both were purchased in Iowa (PV=18.3 vs 65.9 meq/kg); Maseca much less than Pioneer even though both were purchased in Texas (PV=63.4 vs 14.2 meq/kg); and Quaker much less than Maseca, even though both were purchased in Arizona (PV=22.8 vs 48.2 meq/kg). These suggest that the variation observed in Table II is the result of more than turnover rate.

The Quaker masa had uniformly low levels of oxidation at both locations (18.3 meq/kg in Iowa and 22.8 meq/kg in Arizona). On the other hand, wide variation was observed in Maseca (PV=65.7 meq/kg in Iowa; 48.2 meq/kg in Arizona; 34.9 meq/kg in Sonora, México; and 14.2 meq/kg in Texas). These data suggest that Quaker has a very effective distribution system where their products pass through the distribution system faster than Maseca products.

Storage Study

Chemical Composition. The chemical compositions of the two freshly produced Mexican dry corn masa flours used in the shelf-life study are shown in Table I. Both fresh corn masa flours (immediately after sampling from the production lines)

were similar in compositions to those previously reported (Gomez et al 1987) and had low initial lipid oxidation (i.e., CD 0.38-0.78% and PV 4-14 meq/kg of oil).

Reproducibility of Tests

General trends in PV, CD and sensory scores of flours and tortillas were quite reproducible (Fig. 1 is a representative example). Masa flours used for rep 1 were about 25 meq/kg of PV lower than rep 2 (Table I) because the flours for rep 2 had to be stored for 20 wk at -24°C prior to commencing the second replicate of storage trials. Even at -24°C, there was significant lipid oxidation in dry corn masa flour during this period. Sensory analysis and PV and CD values of Maseca (Fig. 1) and Minsa showed the same trends in both storage replicates. Rancid sensory scores were often reached when the PVs exceeded 80 meq/kg or CDs exceeded 1.2%, which resulted after 8 wk of storage at 45°C.

PV ANOVA (Table III) showed that the two reps were different at $P < 1\%$. However, the two masa flours (Table III) had similar CD concentrations at $P < 1\%$. The sensory analyses (Table IV) showed that masa flours and storage reps were different at $P < 1\%$. Rep 2 had higher (rancid) sensory scores for both flour and tortillas (Fig. 1).

Effect of Storage Temperature

Storage time and temperature significantly affected lipid oxidation (Fig. 2). Higher storage temperature and longer storage time led to greater lipid oxidation and shorter shelf-life (time to sensory score ≥ 3). PV and CD values of dry corn masa flours stored at 15-25°C (Fig. 3A) steadily increased during the storage period (23 wk), but flours and tortillas prepared from these flours never reached rancid sensory scores (≥ 3). At 35°C (Fig. 3B) rancid sensory scores were reached after 20 wk storage. At 45°C, PV values increased rapidly peaking at week 20, and tortillas sensory rancid scores (≥ 3) were reached after 9 wk of storage. PV and CD values of masa flours stored at 55°C (Fig. 3C) increased slightly over a period of 10 wk at a much lower rate than flours stored at lower temperatures, then leveled off. We attribute this to the rapid breakdown of conjugated dienoic acids and peroxides at 55°C and above.

Sensory analysis was more effective than chemical analysis in distinguishing between levels of oxidation among the storage temperatures as shown by the mean values during the 23-wk storage time (Table V). Sensory scores increased ($P \leq 1\%$) as storage temperature increased. Flours and tortillas made from flours stored at 55°C were obviously rancid (sensory score ≥ 3) after 1 wk of storage (Table V and Fig. 2).

Lipid oxidation in dry corn masa flour was more rapid as temperature increased as indicated by increasing values of constant rate reaction, k (Tables VI

and VII). Lipid oxidation of dry corn masa flours, using 10 wk storage time, could be described by first-order kinetic functions $dC/dt=kC(1-C/C_{max})$ (Labuza 1982), where C is the concentration of the total oxidation products, C_{max} is the maximum value of parameter C at the end of the lipid oxidation process, k is the reaction rate constant, and t is the time in wk (Figs. A1-A4).

The larger the k value, the faster the formation of oxidization products. For instance, the k value for Maseca flour stored at T 55°C was 0.040 wk^{-1} and the time to reach rancid sensory score was only 1 wk; while at T 15°C, k was 0.015 wk^{-1} and the time to reach rancid score was 54 wk (Table VI). The k value for Minsa flour stored at 55°C was 0.028 wk^{-1} and the time to reach rancid score was less than 1 wk; whereas at 15°C the k value was 0.009 wk^{-1} and the time to reach rancid score was 44 wk (Table VII). Fritsch and Gale (1977) reported k values for corn flakes to be 0.8 at 55°C and 0.07 at 21°C. Thus, dry corn masa is much more prone to oxidation than corn flakes, due that germs are present in dry corn masa flours.

The shelf-life of Maseca dry corn masa flour was 24 wk (6 mo) and 22 wk (5.5 mo) for Minsa when stored at 25°C. Each 10°C increase in storage temperature reduced the shelf-life of dry corn masa flour by almost one-half (Tables VI and VII).

Energy of activation (E_a) and Q_{10} values of flours and tortillas calculated from Arrhenius plots of lipid oxidation for both dry corn masa flours are shown in

Fig. 4. Low Q_{10} s (tortillas, 1.75; and flours, 1.58) and E_a (tortillas, 7.4; and flours, 10.4 kcal/mol °K) as found from the analysis of time to reach rancid score were characteristic those reported by Labuza (1982) Q_{10} generally ranged from 1.5 to 2.0, and E_a from 10 to 25 kcal/mol °K for lipid oxidation of dry cereals. Tortillas required less energy (7.4 kcal/mol °K) to break hydroperoxides into off-flavor and odor products than flours (10.4 kcal/mol °K) (Fig. 4). Therefore, tortillas reached rancid scores faster than flours (Tables VI and VII).

Comparison of Masa Flours

Comparisons of lipid oxidation development at 15 and 45°C of Maseca and Minsa are shown in Figs. 5 and 6, respectively. Minsa flour stored at 45°C developed rancid off-flavors in a shorter period (6 wk) than did Maseca flour (10 wk). PV ANOVA (Table VIII) showed that the flours and storage temperatures and times were different at $P < 1\%$. However, CD ANOVA showed that lipid oxidation of Maseca and Minsa were significantly affected ($P < 1\%$) by storage temperatures and storage times.

Sensory analysis (Table IX) showed that dry corn masa flours were not different at $P < 1\%$; but, tortillas scores showed that the flours and storage time were different at $P < 1\%$. Panelists perceived off-flavors of lipid oxidation in tortillas more readily than in flours; because, cooked tortillas made from rancid flour had extremely rancid aftertastes, which were quite easy to detect.

Each 10°C increase in temperature reduced the shelf-life of dry corn masa flours by about one-half (Tables VI and VII). For instance, Maseca flour stored at 15°C developed rancid off-flavors after 54 wk (12.5 mo) (Table VI) and Minsa reached rancid scores after 44 wk (10 mo) (Table VII). Mean shelf-lives of both dry corn masa flours were about 23 wk (5.8 mo) when stored at 25°C. Differences in shelf-lives for the two flours stored at 15°C may be due to the fact that Minsa had a higher initial PV than Maseca, even though they were processed at the same time. Reasons for these differences are not yet clearly understood, but might be due to differences in the storage condition and age of corn used to make the flours.

Correlation Between Chemical Analysis and Sensory Scores

Storage temperature greatly affected the correlation of CD and PV with sensory scores of flours and tortillas (Fig. 7). The best correlations of CD and PV values with sensory scores occurred in flours stored at 45°C; however, correlations of PV and CD with tortilla or flour sensory scores were generally poor for both masa flours.

Figure 8 shows correlation where PV and CD values of both masas (where all temperatures were combined) with the data pool of sensory scores. CD values showed a significant correlation of 0.42 with the sensory scores of flours and 0.38 with the sensory scores of tortillas. PV values had a significant correlation of 0.46

with the sensory scores of flours and 0.35 with the sensory scores of tortillas.

Thus, PV and CD values were poor predictors of sensory perception of rancidity in dry corn masa flour.

CONCLUSIONS

A retail marketplace survey showed that the oil of dry corn masa is typically quite oxidized. Rancidity of dry corn masa flours was greatest during the summer and least during the winter. Shelf-life of dry corn masa flour was 5.8 mo when stored at 25°C. Each 10°C increase in temperature reduced the shelf-life of dry corn masa flour by about one-half. PV and CD were not good predictors of sensory perception of rancidity in dry corn masa flours. Sensory evaluation of tortillas was a much more sensitive measure of rancidity than sensory analysis of flours. This study illustrated the need for methods to measure and control lipid oxidation of dry corn masa flour.

LITERATURE CITED

- AMERICAN OIL CHEMISTS SOCIETY. 1990. Approved Methods of the AOCS, 5th ed. Method cd8-53 approved 1989, ti 1a-64 approved 1989. The Association: Champaign, IL.
- BRESSANI, R. 1990. Chemistry, technology, and nutritive value of maize tortillas. *Food Rev. Int.* 6:225-264.
- CARRILLO-PEREZ, E., SERNA-SALDIVAR, S. O., and ROUZAUD-SANDEZ, O. 1989. Effect of storage conditions and packaging materials on the physicochemical, microbiological and sensory properties of corn dry masa flour. *J. Food Proc. Preserv.* 13:335-353.
- FRITSCH, C. W., and GALE, J. A. 1977. Hexanal as a measure of oxidative deterioration in low fat foods. *J. Am. Oil Chem. Soc.* 54:225-231.
- GOMEZ, M. H., ROONEY, L. W., and WANISKA, R. D. 1987. Dry corn masa flours for tortilla and snack production. *Cereal Foods World* 32:372-377.
- LABUZA, T. P. 1982. Shelf-life of pasta products. Pages 119-129 in: *Shelf-life Dating of Foods*. Food and Nutrition Press, Inc.: Westport, CN.
- MOSKOWITZ, H. R. 1980. Utilitarian benefits of magnitude estimation scaling for testing product acceptability. Pages 11-18 in: *ASTM Special Technical Publication 773*. American Society for Testing Materials: Philadelphia, PA.
- NAWAR, W. W. 1985. Lipids. Pages 138-169 in: *Food Chemistry*. O. R. Fennema, ed. Marcel Dekker: New York and Basel.
-

POKORNY, J. 1987. Major factors affecting the autoxidation of Lipids, Pages 141-206 in: Autoxidation of Unsaturated Lipids. H. W. S. Chang, ed. Academic Press: London, Orlando, San Diego.

SAS INSTITUTE, INC. 1989. SAS/STAT User's Guide, version 6, 4th ed., Vol. 1, and 2. The Institute: Cary, NC.

SANDERS, T. A. B. 1989. Nutritional aspects of rancidity. Pages 125-140 in: Rancidity in Foods. J. C. Allen and R. J. Hamilton, eds. Elsevier Applied Science: London and New York.

STONE, H., and SIDEL, J. 1985. Introduction to sensory evaluation. Pages 66-94 in: Sensory Evaluation Practices. 2nd ed. Academic Press, Inc.: San Diego, New York.

TABLE I
Properties of Two Commercial Dry Corn Masa Flours from México

Property	Maseca	Minsa
Moisture (%) ¹	9.00	10.40
Fat (%) ¹	4.21	4.32
Protein (%) ^{1,2}	9.01	9.09
Ash (%) ¹	1.52	1.61
pH	7.52	7.41
Initial PV (meq/kg)		
Rep 1 ³	3.7	14.0
Rep 2	31.3	30.0
Initial CD (%)		
Rep 1	0.38	0.59
Rep 2	0.59	0.78
Initial sensory score ⁴		
Flours		
Rep 1	1.6	1.3
Rep 2	1.6	1.5
Tortillas		
Rep 1	1.5	1.3
Rep 2	1.5	1.4

¹ All values are means of three replicates and expressed on dry matter basis, except moisture.

² %N x 6.25.

³ Storage period replicate.

⁴ 1=not rancid and 5=extremely rancid.

TABLE II
Peroxide Values (meq/kg) of Dry Corn Masa Flours from
México and the United States

Masa Source¹	Winter '92	Spring '92	Summer '92	Fall '92	Winter '93	Mean LSD=5.8²
mas	8.4	33.8	59.5	44.0	28.7	34.9 ^d
min	55.7	66.0	59.9	66.3	41.2	57.8 ^b
maa	38.9	98.2	82.6	13.6	7.7	48.2 ^c
qua	9.5	25.9	32.7	28.8	17.1	22.8 ^e
pio	65.3	38.6	56.5	79.7	76.9	63.4 ^{ab}
mat	4.8	22.7	10.6	13.4	19.0	14.2 ^f
mai	77.0	96.4	53.7	63.6	38.9	65.9 ^a
qui	5.8	20.0	12.5	29.4	23.9	18.3 ^{ef}
Mean LSD=4.6 ²	33.2 ^c	50.2 ^a	45.8 ^{ab}	42.4 ^b	31.7 ^c	

¹ Maseca mas=purchased from México, maa=purchased from Arizona, mat=purchased from Texas, and mai=purchased from Iowa; Minsa min=purchased from México; Quaker qua=purchased from Arizona and qui=purchased from Iowa; and Pioneer pio=purchased from Texas.

² Least significant difference (P<5%, DF=120).

^{a,b,c,d,e,f} Means with the same superscript within the last column or bottom row are not significantly different at P<5%.

TABLE III
ANOVA of Chemical Analyses of Dry Corn Masa Flours¹

Source of Variation	Degrees of Freedom	Mean Sum of Squares	F Value ²
Peroxide Value			
FL ³	1	6446.4	16.3**
T ⁴	4	478.9	0.4ns
FL*T*Rep	5	40759.9	20.6**
Rep ⁵	1	242155.7	611.0**
T*Rep ⁶	4	278770.2	49.7 **
Conjugated Dienoic Acids			
FL ³	1	0.1	0.2ns
T ⁴	4	1.6	1.2ns
FL*T*Rep	5	11.9	8.2**
Rep ⁵	1	101.6	46.7**
T*Rep ⁶	4	11.9	1.4ns

¹ Degrees of freedom (DF); DF_{Total}=819, DF_{Model}=409, DF_{Error}=410.

² * 5%, significance level; ** 1%, significance levels; and ns, not significant.

³ Flour.

⁴ Temperature.

⁵ Storage period replicate.

⁶ FL*T*Rep used as the error term.

TABLE IV
ANOVA of Sensory Analyses of Dry Corn Masa Flours¹

Source of Variation	Degrees of Freedom	Flours		Tortillas	
		Mean Sum of Squares	F Value ²	Mean Sum of Squares	F Value ²
FL ³	1	0.3	3.2**	0.9	8.3**
T ⁴	4	4.8	4.2**	3.3	7.4**
FL*T*Rep	5	1.5	3.3**	0.9	1.4ns
Rep ⁵	1	4.6	15.9**	8.2	72.3**
T*Rep ⁶	4	0.8	0.7ns	3.6	1.9ns

¹ Degrees of freedom (DF); DF_{Total}=819, DF_{Model}=409, DF_{Error}=410.

² * 5%, significance level; ** 1%, significance levels; and ns, not significant.

³ Flour.

⁴ Temperature.

⁵ Storage period replicate.

⁶ FL*T*Rep used as the error term.

TABLE V
Effect of Temperature on Mean Values of Chemical and Sensory
Analyses of Dry Corn Masa Flours¹

Temperature °C	Chemical Analyses		Sensory Analyses ²	
	PV (meq/kg)	CD (%)	Flours	Tortillas
15	64.7 ^{cb}	0.96 ^c	1.75 ^e	1.90 ^d
25	79.9 ^{ab}	1.18 ^{cb}	2.24 ^d	2.43 ^e
35	94.8 ^a	1.48 ^{ab}	2.54 ^c	2.59 ^c
45	93.0 ^a	1.62 ^a	3.10 ^b	3.02 ^b
55	44.8 ^c	1.40 ^{ab}	3.90 ^a	4.03 ^a
LSD ³	25.6	0.42	0.11	0.13

¹ Mean values over 23-wk storage.

² Means of subjective evaluation in which 1=no rancidity, 2=slightly rancid, 3=rancid, 4=very rancid, and 5=extremely rancid. ^{a,b,c,e} Means with the same superscript within a column are not significantly different at P<5%.

³ Least significant difference (P<5%, DF_{chemical}=204, DF_{sensory}=59, Fl*T*Rep used as the error term, Fl=flour, T=temperature, Rep=storage time).

TABLE VI
Rate Constant k, Time to Rancid Score, and Chemical Properties Calculated
from Model Equations for Lipid Oxidation of Maseca Dry Corn Masa Flour¹

Tem. (°C)	kx10 ⁻³ Rate Constant (wk ⁻¹)	Model Equation ²	r ²	Time to Rancid (wk)	Values at Rancid Score	
					PV (meq/kg)	CD (%)
Flours						
15	15	y=0.026x + 1.60	0.40	54	-	-
25	19	y=0.051x + 1.78	0.60	24	-	-
35	25	y=0.045x + 2.09	0.17	20	130	1.91
45	41	y=0.102x + 1.92	0.74	10	89	1.34
55	40	y=0.138x + 2.84	0.93	1	37	0.68
Tortillas						
15	32	y=0.051x + 1.28	0.53	33	-	-
25	34	y=0.088x + 1.61	0.71	16	88	1.24
35	44	y=0.077x + 1.87	0.80	15	105	2.28
45	52	y=0.130x + 1.88	0.97	8	83	1.38
55	61	y=0.199x + 2.37	0.89	3	38	0.63

¹ Model equation using sensory data from the first 10 wk of storage time.

² Y=sensory score and X=storage time in wk.

TABLE VII
Rate Constant k, Time to Rancid Score, and Chemical Properties Calculated
from Model Equations for Lipid Oxidation of Minsa Dry Corn Masa Flour¹

Tem. (°C)	kx10 ⁻³ Rate Constant (wk ⁻¹)	Model Equation ²	r ²	Time to Rancid (wk)	Values at Rancid Score	
					PV (meq/kg)	CD (%)
Flours						
15	9	y=0.038x + 1.33	0.76	44	-	-
25	25	y=0.044x + 2.02	0.29	22	-	-
35	29	y=0.069x + 2.05	0.72	14	-	-
45	24	y=0.061x + 2.47	0.52	9	104	1.61
55	28	y=0.102x + 3.22	0.88	0	89	1.36
Tortillas						
15	23	y=0.079x + 1.47	0.83	19	75	0.99
25	23	y=0.056x + 2.15	0.51	15	96	1.72
35	27	y=0.066x + 2.12	0.52	13	103	1.77
45	46	y=0.124x + 1.98	0.99	8	85	1.43
55	51	y=0.177x + 2.81	0.88	1	78	0.63

¹ Model equation using sensory data from the first 10 wk of storage time.

² Y=sensory score and X=storage time in wk.

TABLE VIII
ANOVA for Chemical Analyses to Test Storage Time
of Dry Corn Masa Flours¹

Source of Variation	Degrees of Freedom	Mean Sum of Squares	F Value ²
Peroxide Value			
FL ³	1	1.35	9.45**
T ⁴	4	74.70	130.91**
FL*T*ST	204	35.83	1.23*
ST ⁵	40	82.35	14.43**
T*ST	160	24.58	1.08ns
Conjugated Dienoic Acids			
FL ³	1	0.22	3.07ns
T ⁴	4	21.76	5.92**
FL*T*ST	204	14.36	0.98ns
ST ⁵	40	80.58	28.11**
T*ST	160	10.38	1.91ns

¹ Degrees of freedom (DF); DF_{Total}=819; DF_{model}=409; DF_{Error}=410.

² * 5%, significance level; ** 1%, significance level; and ns, not significant.

³ Flour.

⁴ Temperature.

⁵ Storage time.

TABLE IX
ANOVA for Sensory Analyses to Test Storage Time
of Dry Corn Masa Flours¹

Source of Variation	Degrees of Freedom	Flours		Tortillas	
		Mean Sum of Squares	F Value ²	Mean Sum of Squares	F Value ²
FL ³	1	0.1	1.0ns	5.4	55.4**
T ⁴	4	130.7	417.7**	122.2	312.1**
FL*T*ST	59	4.7	1.2ns	5.9	1.0*
ST ⁵	11	15.5	18.0**	28.2	26.1**
T*ST	44	8.8	2.6**	7.9	1.8**

¹ Degrees of freedom (DF); DF_{Total}=239; DF_{Model}=119; DF_{Error}=120.

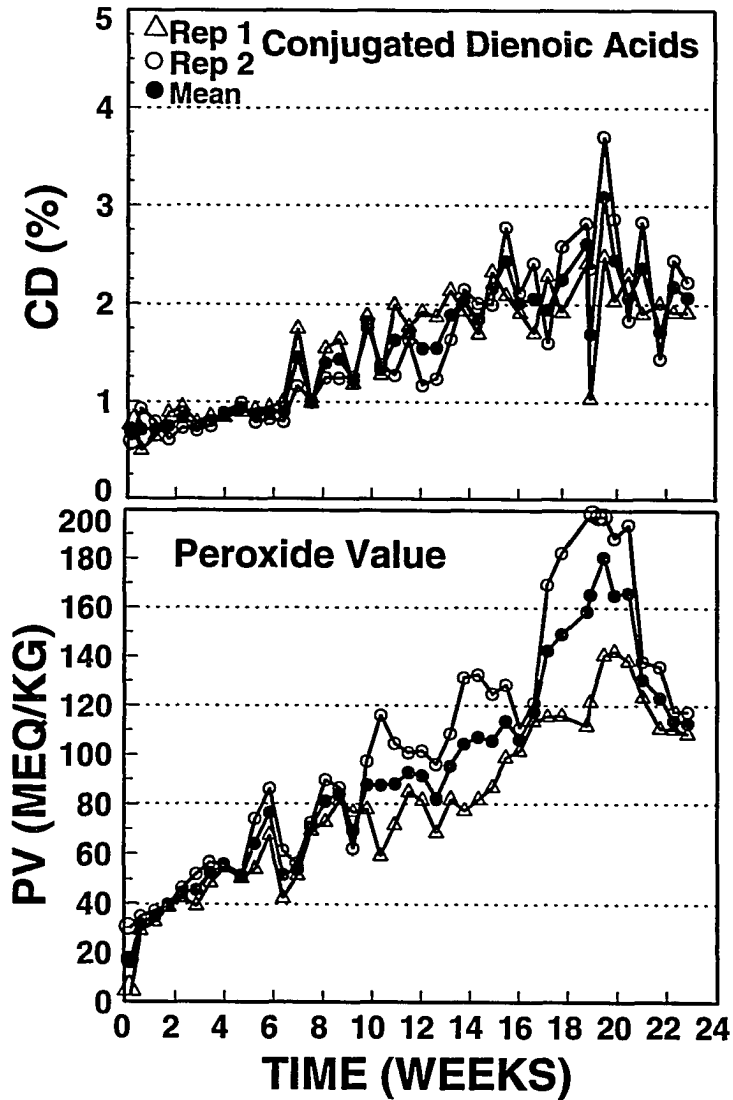
² * 5%, significance level; ** 1%, significance level; and ns, not significant.

³ Flour.

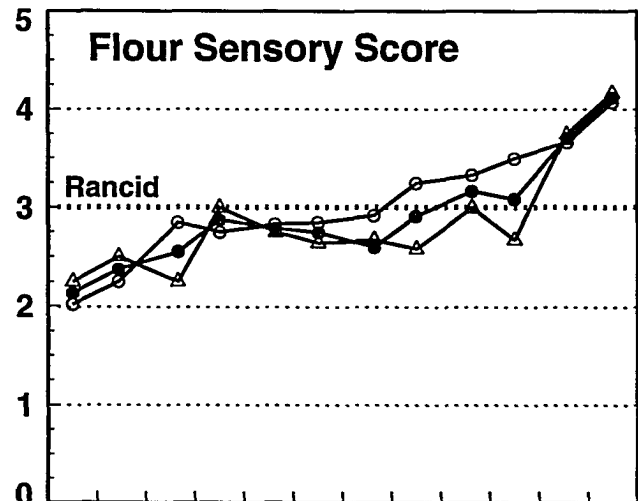
⁴ Temperature.

⁵ Storage time.

Fig. 1 Reproducibility of tests used for measuring lipid oxidation of Maseca dry corn masa flour
(stored at 45°C).



SENSORY SCORE



SENSORY SCORE

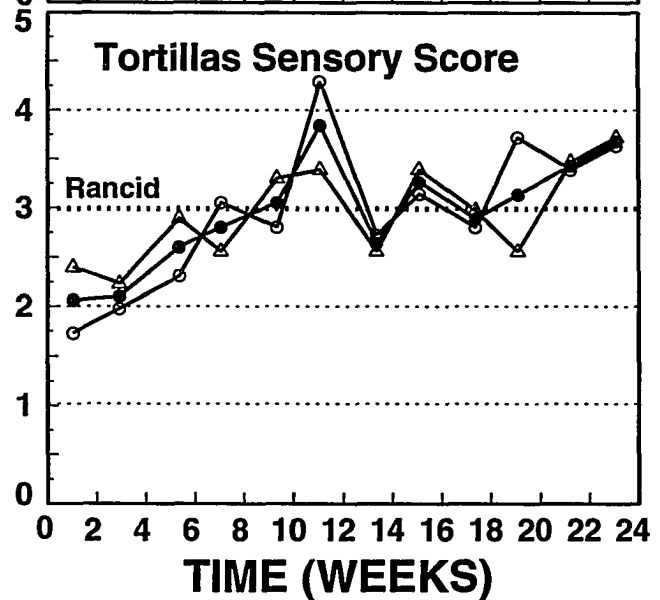
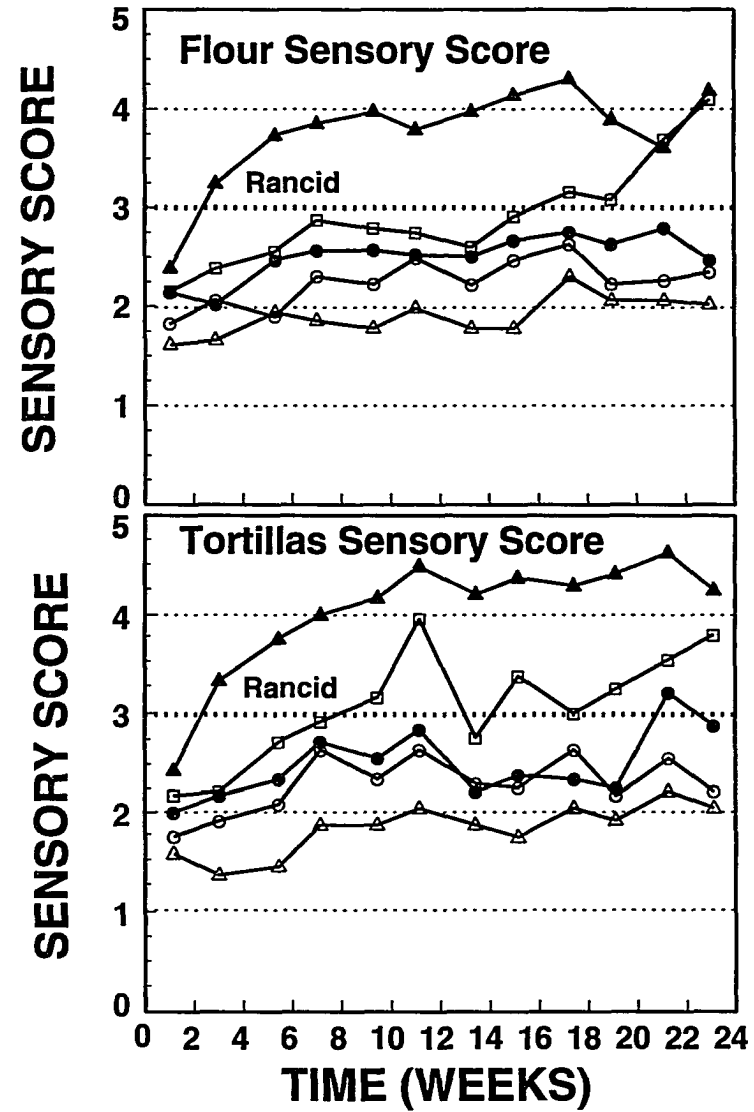
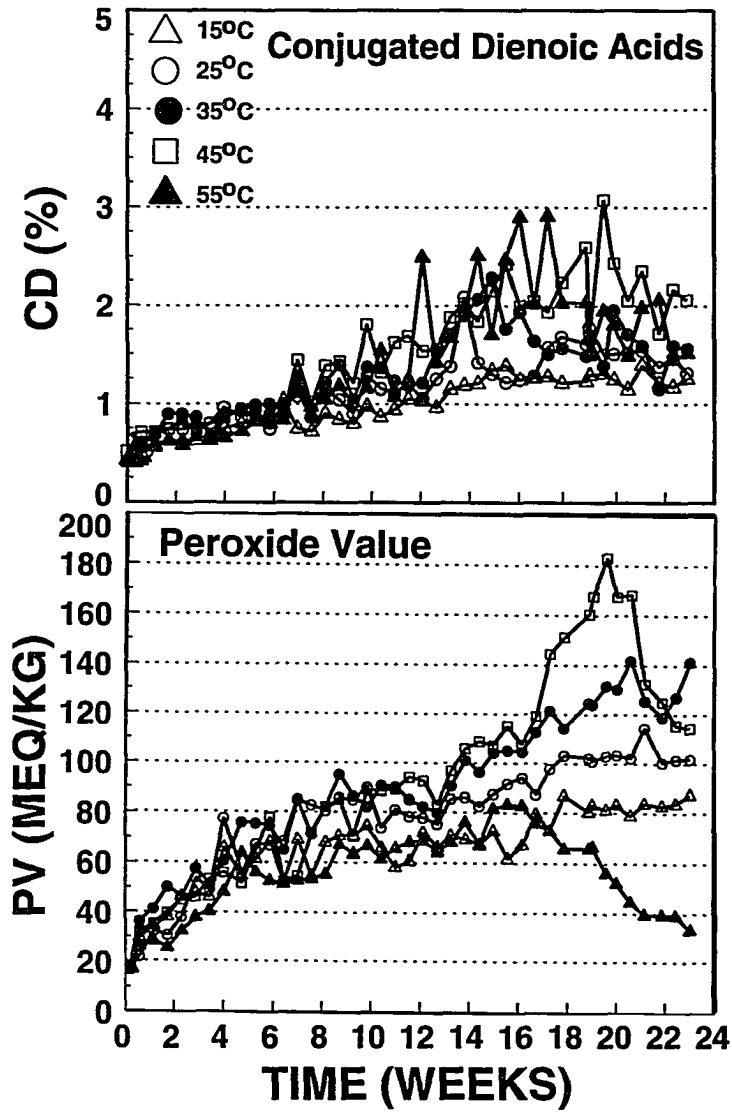


Fig. 2 Effect of temperature on lipid oxidation of dry corn masa flour (Maseca).



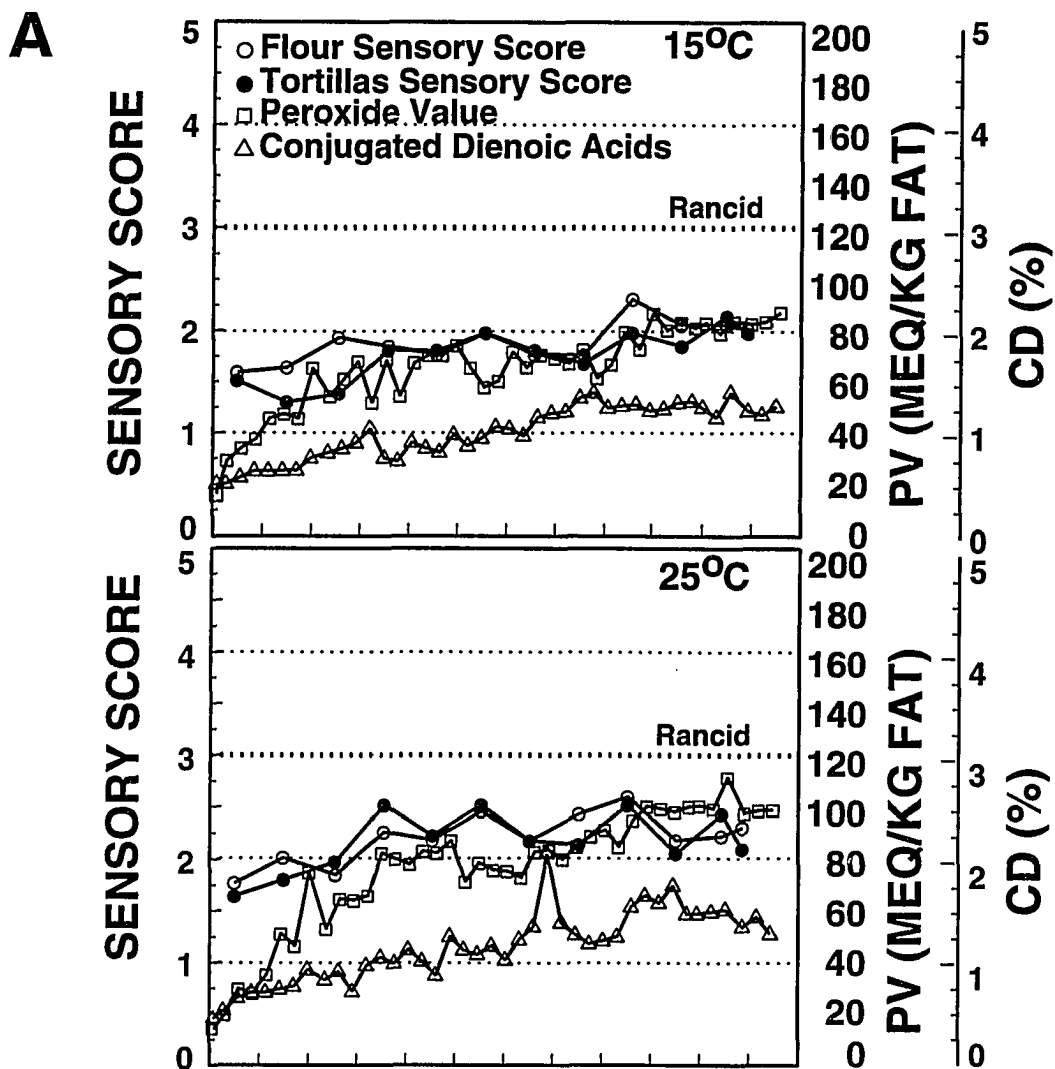


Fig. 3 Effect of temperature on lipid oxidation of dry corn masa (Maseca)

(A, 15 and 25 °C; B, 35 and 45°C; and C, 55°C).

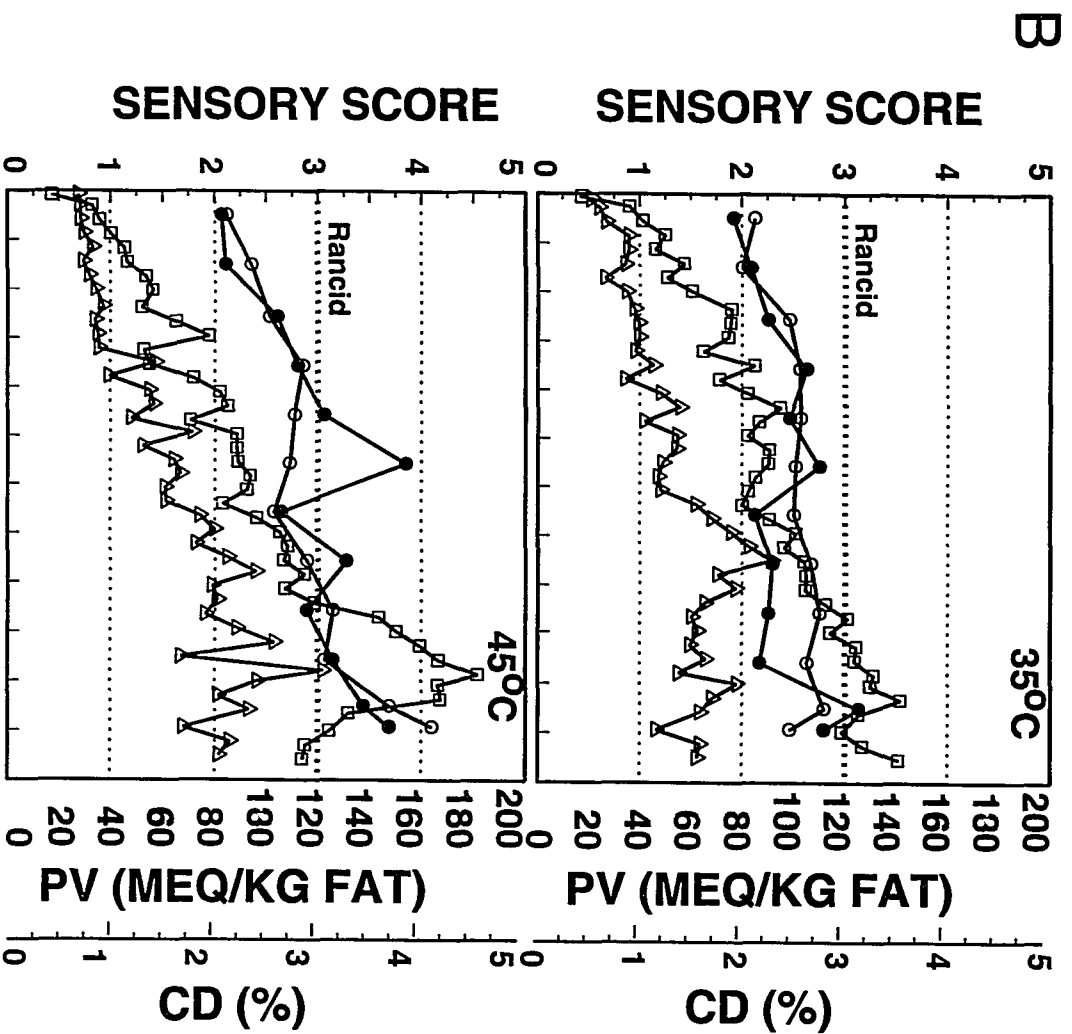


Fig. 3 Continuation.

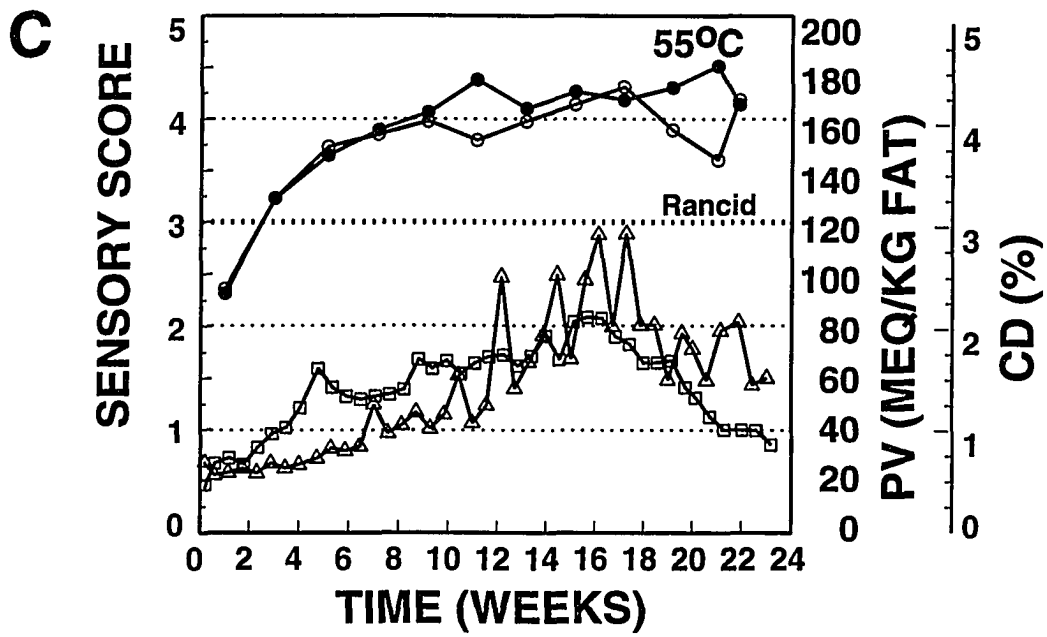


Fig. 3 Continuation.

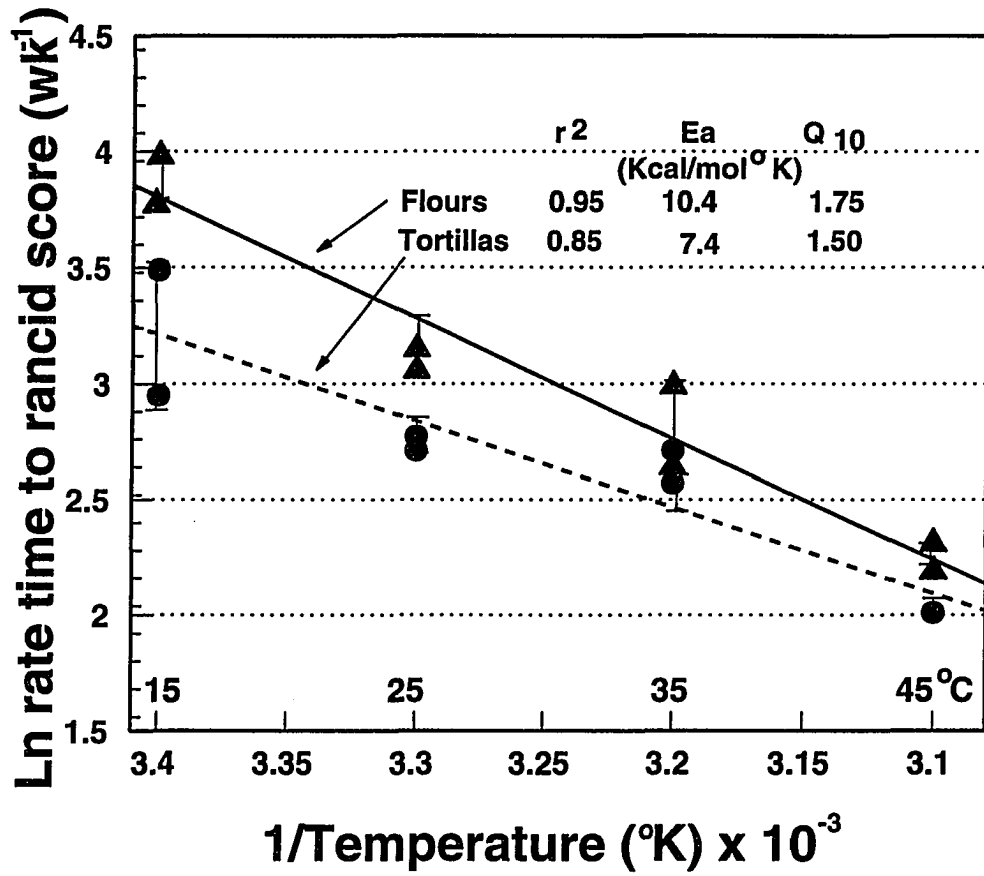


Fig. 4. Arrhenius plots of lipid oxidation in dry corn masa flour.

Fig. 5 Comparison of lipid oxidation properties of two commercial dry corn masa flours stored at 15°C.

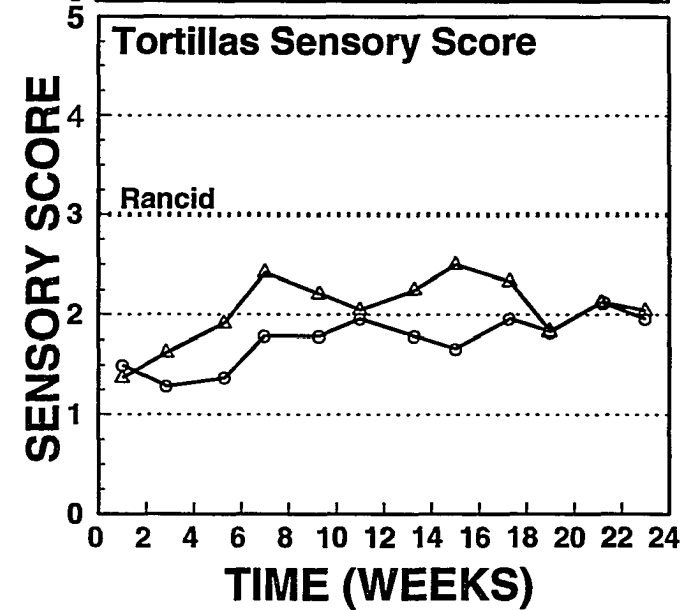
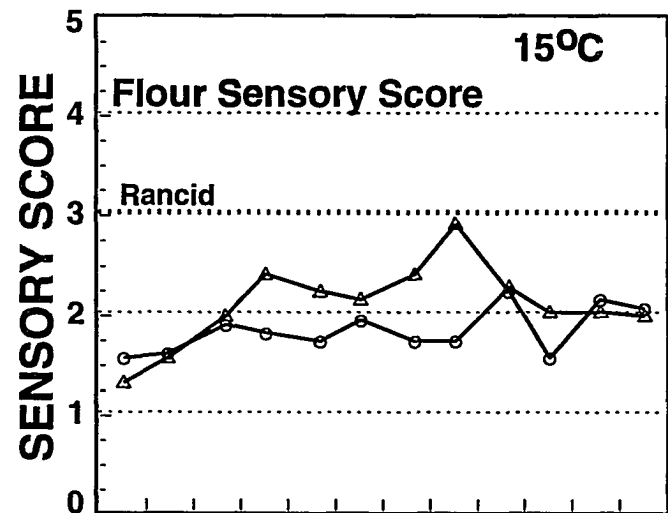
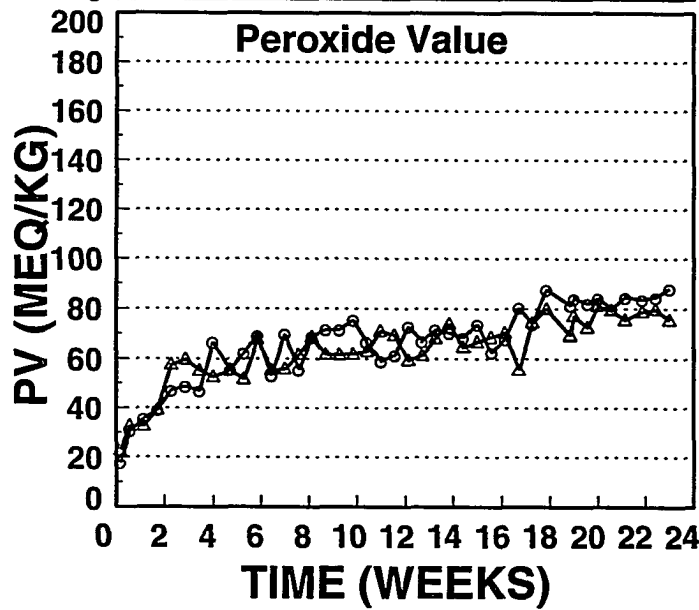
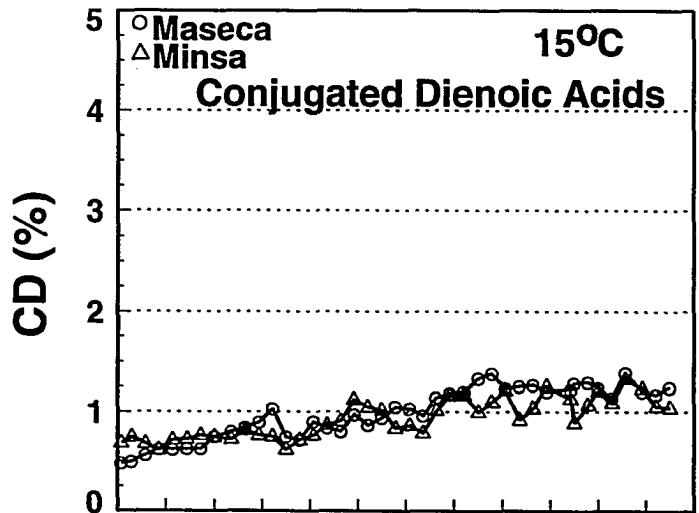


Fig. 6 Comparison of lipid oxidation properties of two commercial dry corn masa flours stored at 45°C.

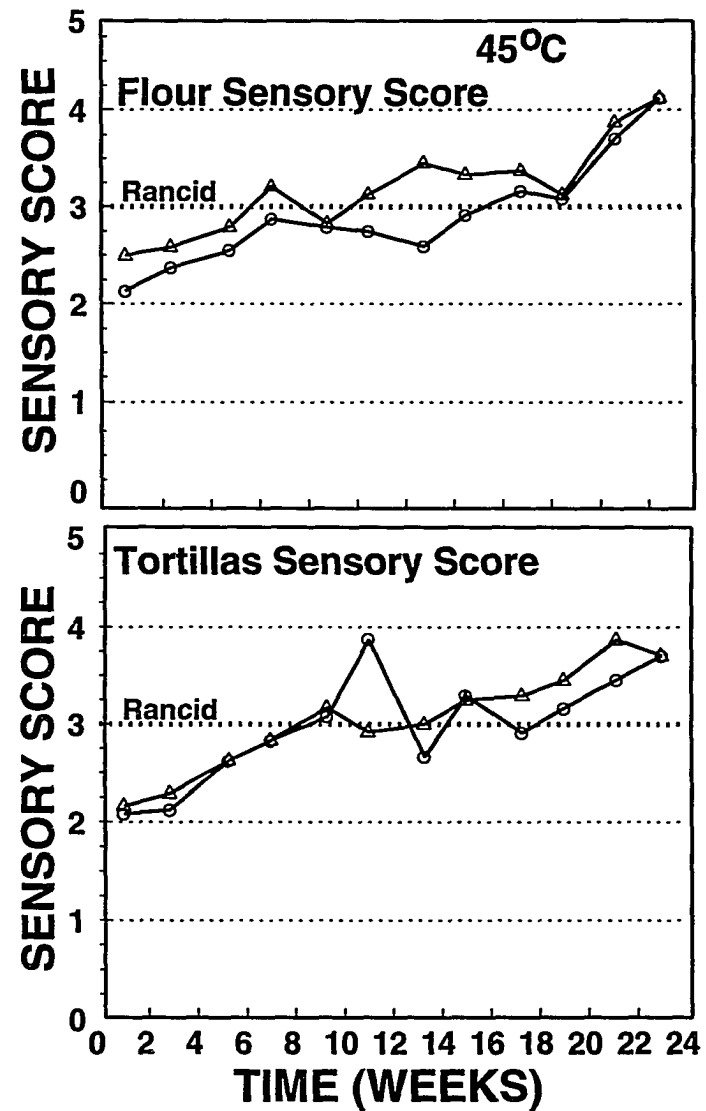
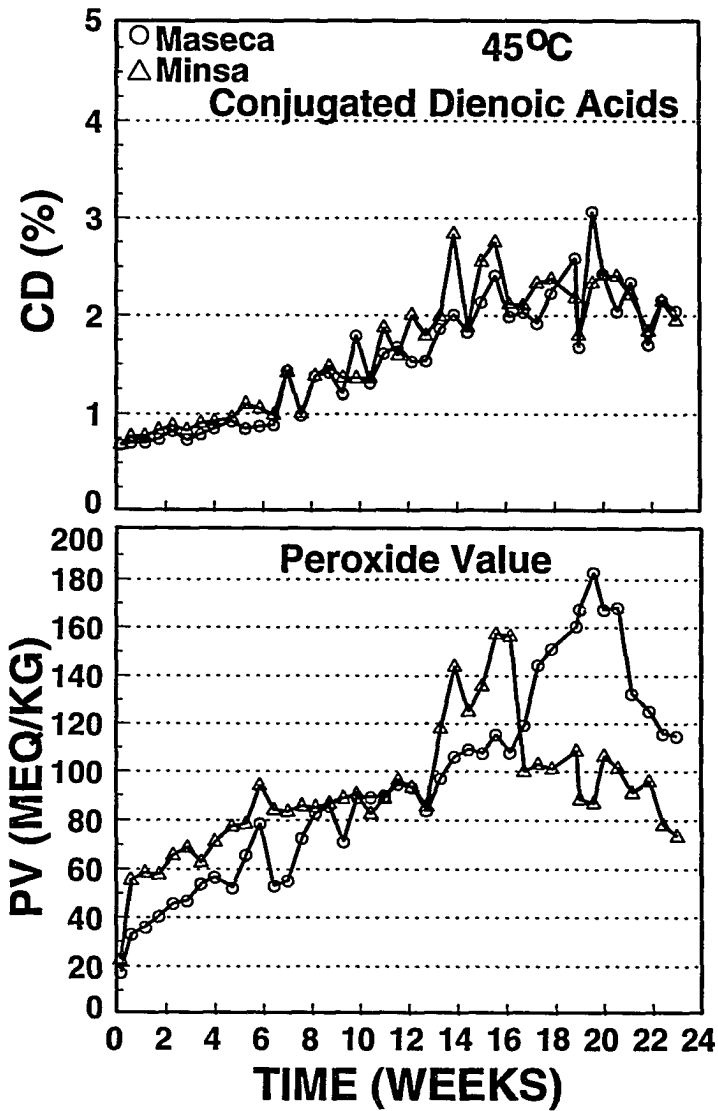
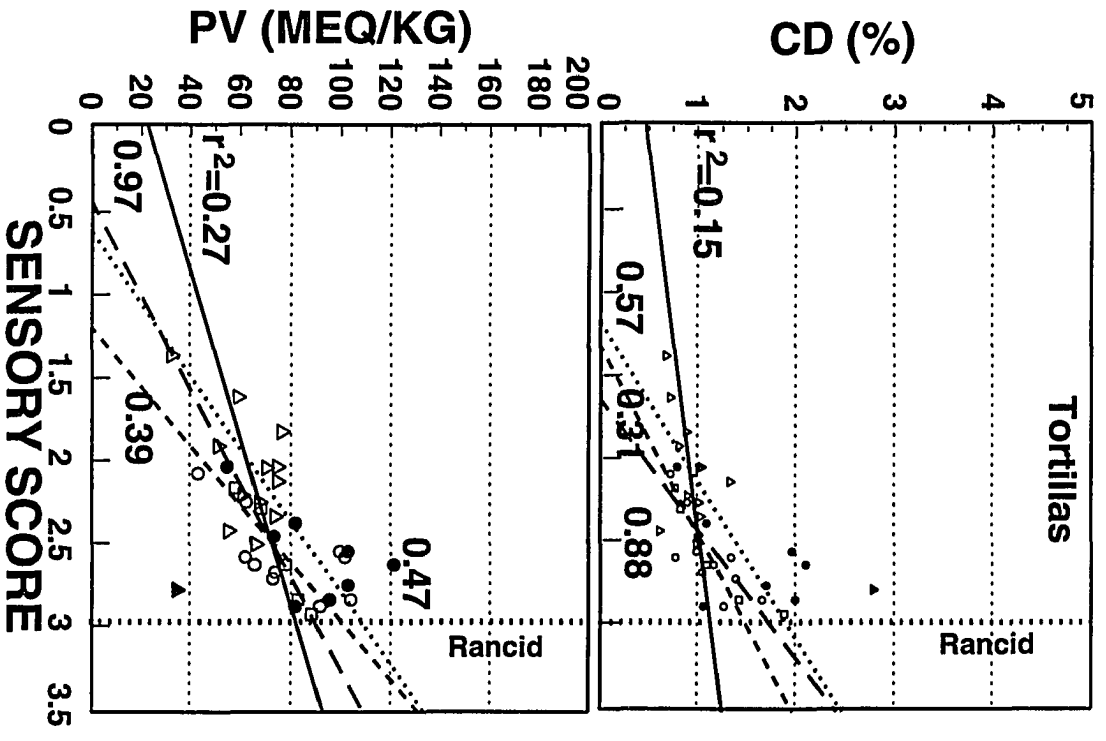
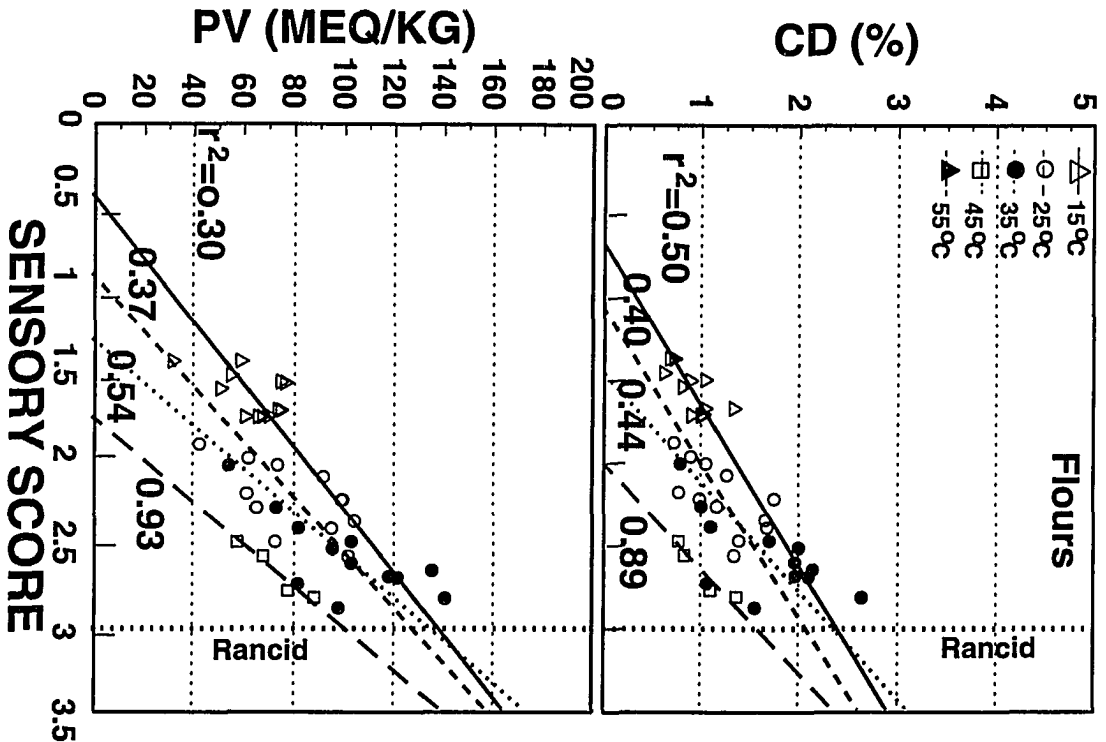


Fig. 7 Correlation of sensory data (scores ≤ 3) with chemical properties of dry corn masa flour (Maseca) at various temperatures.



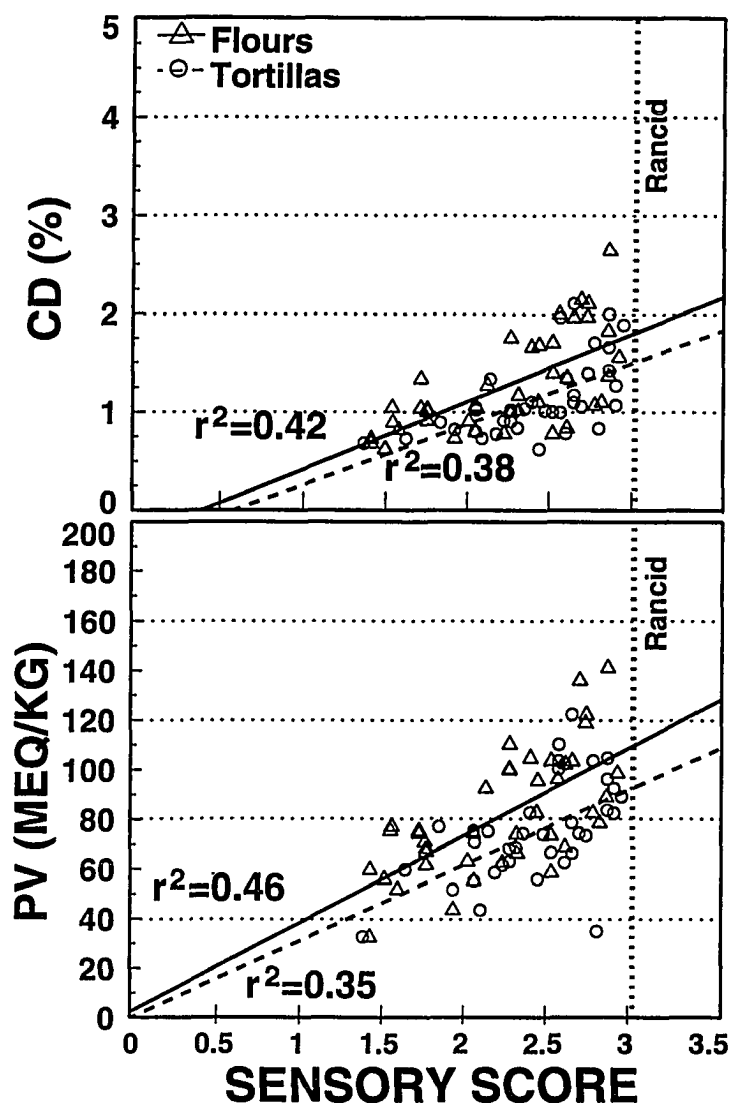


Fig. 8 Correlation of chemical and sensory tests grouped into flours and tortillas, regardless storage temperature and manufacturers.

PAPER II

**ROLE OF OIL ON PHYSICAL PROPERTIES OF DRY CORN MASA FLOUR
AND SENSORY CHARACTERISTICS OF CORN TORTILLAS**

**Role of Oil on Physical Properties of Dry Corn Masa Flour and
Sensory Characteristics of Corn Tortillas¹**

**Reyna Luz Vidal-Quintanar², Lawrence A. Johnson^{2,3,4},
and Jane A. Love³**

¹ Journal Paper J- of the Iowa Agriculture and Home Economics Experiment Station, Ames, IA, 50011. Project 0178. Research supported by the Center for Crops Utilization Research and the Iowa Agriculture and Home Economics Experiment Station.

² Graduate Research Assistant and Professor-in-Charge, respectively, Center for Crops Utilization Research, Iowa State University, Ames, IA, 50011.

³ Professor and Associate Professor, respectively, Department of Food Science and Human Nutrition, Iowa State University, Ames, IA, 50011.

⁴ To whom correspondence should be addressed.

ABSTRACT

Commercial and laboratory-made (lab-made) dry corn masa flours were extracted with hexane and each of the near fat-free flours were reconstituted with 2 and 4% w/w commercial corn oil (near fat-free, 50 and 100% reconstitution, respectively) to determine the impact of oil on dough properties and sensory characteristics of corn tortillas. Commercial and lab-made flours had very similar chemical compositions, physical properties, and sensory characteristics, indicating that the lab method of preparing dry corn masa adequately simulated commercial dry corn masa. Sensory analysis of masa flour from both processes showed the same patterns. The absence of oil did not affect ($P < 5\%$) dough stickiness or tortillas rollability. The absence of oil significantly and adversely affected the typical corn flavor of tortillas. Oil significantly improved tortilla firmness and chewiness. The important contributions of oil to flavor, chewiness and firmness of corn tortillas made it unfeasible to extend shelf-life of dry corn masa flour by extracting the oil.

INTRODUCTION

Pure food lipids are nearly odorless. However, lipids are precursors to flavor components produced by a wide range of reactions when foods are cooked. Aldehydes and ketones are the main volatiles from oxidation of lipids and can cause painty, fatty, metallic, papery, and candle-like flavors in foods. However, many desirable flavors in cooked and processed foods originate from modest concentrations of lipid-derived compounds (Nawar 1985). Little progress has been made in preventing undesirable off-flavors in foods that contain unsaturated fatty acids, mainly because of the large variety of products generated during lipid oxidation, interactions of lipid decomposition products with one another, and with other nonlipid food components.

Vidal-Quintanar et al (1993) reported that autoxidation of oil causes off-flavors and off-odors of dry corn masa flours, which reduce consumer acceptability of the corn tortillas. They reported that dry corn masa flour has a shelf-life of 23 wk when stored at 25°C, but only 9 wk at 45°C.

Undesirable flavors and odors from oxidation of oil in dry corn masa flours would likely be prevented by extracting the oil or by degerming the nixtamal. But, the effects of oil on dough properties and sensory characteristics of corn tortillas are unknown. However, in wheat flour doughs, MacRitchie and Grass (1973) found that neutral lipids did not affect rheological properties of the dough. Segushi and Matsuki (1977) and Segushi (1984) reported that lipids reduce interactions

between proteins and partially gelatinized starch of wheat dough by forming slip planes. Therefore, the objectives of the present paper were to develop a laboratory procedure for preparing dry corn masa flour to study the function of oil in tortillas production and to determine the effects of corn oil on physical properties and sensory characteristics of corn tortillas.

MATERIAL AND METHODS

Laboratory Process for Preparing Dry Corn Masa Flours

The procedure used for preparing laboratory-made (lab-made) dry corn masa is shown in Fig. 1. White corn (Pioneer 3281, Pioneer Hi-Bred International, Inc., Johnston, IA) and 1% calcium hydroxide (Fisher Scientific, Fair Lawn, NJ) were mixed in a 1:2 corn:water ratio and then cooked by the traditional village-cooking method at 95°C for 95 min (Khan et al 1982). The cooked corn (nixtamal) was soaked overnight at room temperature, washed with 1.5 L tap water three times, and then hand-milled using an Estrella No. 1 hand mill (Corona Ind., Monterrey, NL, México). The masa was dried in a convection oven for 6 hr at 95 ± 3°C. Finally, the dry corn masas were ground using an impact mill (Model D, Fitzpatrick hammermill, Fitzpatrick Co., Chicago, IL) equipped with a 60-mesh screen.

Near Fat-Free Flour Preparation

Neutral lipids was extracted from commercial (Maseca, Azteca Milling Co., Plainview, TX) and lab-made dry corn masa flours with hexane for 5 hr in a Soxhlet extractor (Precision Instrument Co., Chicago, IL) (Fig. 2). After oil extraction, each flour was divided into three lots. Lot 1 from each flour was air desolventized in a hood at room temperature for 3 days. Then, each of the near fat-free flours was vacuum dried in a rotary evaporator for 30 min at 45°C and 30 rpm.

Addition of Commercial Corn Oil to Near Fat-Free Corn Dry Masa Flour

Commercial corn oil (Mazola Corn Oil, Best Foods CPC International Inc., Englewood Cliffs, NJ) was added to lots 2 and 3 of both sources of near fat-free dry corn masa flours (commercial and lab-made). Corn oil (2 and 4% flour weight representing 50 and 100% reconstitution levels, respectively) was dissolved in sufficient hexane (about 1 L) to cover the flour (Fig. 2). Flour-oil slurries were mixed and the bulk solvent was evaporated over a 10-min period. The reconstituted flours were air-dried for 3 days in a hood to evaporate final traces of solvent. Any residual solvent was evaporated in a rotary evaporator under water aspirator vacuum (30 min at 45°C and 30 rpm).

Proximate Analyses and pH

Moisture, fat, ash, and protein (%N x 6.25) contents were determined in triplicate for each treatment using AOAC methods (AOAC 1988). pH was determined using 10 g of dry corn masa flour and 100 ml of distilled water. The slurry was mixed by a magnetic stirring bar for 30 min at speed 7 on a stir-plate prior to measuring pH.

Particle Size Distribution

Mean particle size (D_{gw}) and the geometrical standard deviation of sample estimate by mass (S_{gw}) were determined using 100 g of dry corn masa flour

(ASAE 1990). US standard sieves (50, 70, 100, and 200 mesh) with nominal sieve openings of 0.297, 0.210, 0.149, and 0.074 mm, respectively, were used in a Tyler Ro-tap sieve shaker (T-673 Model B, Combustion Engineering Inc., Mentor, OH). The mass of sample on each screen was determined after 10 min of shaking.

Color Measurement

Colors of flours were measured under fluorescent lighting using the 10° standard observer. Samples were evaluated, where 'L' measured lightness, 'a' measured red (+) and green (-), and 'b' measured yellow (+) and blue (-) coordinates, by using a Hunterlab digital color difference meter (model JB 1201, Hunter Associates Laboratory, Reston, VA). Tiles 'L'=79.21, 'a'=-0.61, and 'b'=22.78 were used for standardization. The total color difference (E value) between the sample color and the corresponding difference tile was calculated as $E=(L^2+a^2+b^2)^{1/2}$ (Hunter 1982).

Dough Properties

Adhesiveness. Adhesiveness or stickiness is defined as the work needed to overcome the adhesive forces of the masa. A stickiness device was constructed following the model of Ramirez et al (1993). Masa dough was made from 200 g of dry corn masa flour and sufficient water to reach the moisture content for optimum consistency ($57 \pm 1\%$ for commercial and $64 \pm 1\%$ for lab-made). A 15 x 4 cm

dough cylinder was formed, placed between the two bars, and compressed. The masa was cut in such a way that it made a partial V-shaped cut in the center of the masa block. The length (cm) of the masa held on the upper bar was recorded and expressed as percentage stickiness. Stickiness values between 10-30% are considered to predict acceptable dough texture (Ramirez et al 1993).

Tortilla Rollability. The rollability of corn tortillas was determined by using the procedure of Bedolla et al (1983). Five tortillas were cut into strips 2 x 1 cm wide. Tortilla strips were gently rolled around a 1.5 cm diameter dowel. A subjective used where a score of 1 denoted no breakage, and 5 denoted a high tendency for tortillas to break.

Sensory Characteristics

Selection and training of the sensory panel were carried out as described by Vidal-Quintanar et al (1993). A group of four sets of freshly-cooked tortillas (one set for each treatment) was presented to each panelist in random order under red light. Panelists evaluated each treatment by placing a vertical line across a 15-cm line scale (Appendix A3) anchored at the ends with terms which best described corn flavor, chewiness, and firmness (from no corn flavor, not chewy, and tender/soft to intense corn flavor, chewy and firm, respectively) (Stone and

Sidel 1985). Testing was done in an air-conditioned panel room equipped with individual booths.

Statistical Analysis

Analysis of variance (ANOVA) procedure (SAS Institute, Inc., 1988) was used to analyze the data. Samples were evaluated in two separate randomized complete block designs with four treatments: (1) full-fat commercial dry corn masa flour and commercial dry corn masa with three levels (near fat-free, 50 and 100%) of reconstituted commercial corn oil; and (2) full-fat lab-made dry corn masa flour and lab-made dry corn masa flours with three levels (near fat-free, 50 and 100%) of reconstituted commercial oil. Physical analysis of each treatment was replicated four times, and sensory analysis was replicated three times. When ANOVA revealed significant effects ($P \leq 1$ or 5%), means were separated by using Fisher's Least Significant Difference procedure, LSD ($P < 5\%$).

RESULTS AND DISCUSSION

Proximate Analysis, pH and Mean Particle Sizes

Moisture contents of all lab-made dry corn masa flavors were significantly lower than those of the commercial flours (Tables I and II). The differences in moisture contents were not important as both flour sources were rehydrated to the same dough development level (homogeneous hydration and smoothness).

The protein contents of lab-made dry corn masa flours were slightly lower (8.6%) than those of commercial dry corn masa flours (9.1%), but were typical of the corn hybrid (Pioneer 3281, with 8.8% protein) used for lab-made dry corn masa flour. Analyses of fat contents showed that the desired oil levels were achieved. Ash contents were similar for all treatments (Tables I and II).

The pH values of lab-made dry masa corn flours were higher than that of untreated flour, and were not affected by oil reconstitution (Table II). The pH differences between lab-made and commercial dry corn masa flours were probably due to less washing of the lab-made nixtamal. Hand washing in our lab procedure was less rigorous than what could be accomplished by industrial equipment. pHs of both masa flours were within ranges reported by Gomez et al (1987). Mean particle sizes of the lab-made dry corn masa flours ($D_{gw}=0.21$ mm and $S_{gw}=2.18$) were the same ($P<1\%$) as that of commercial dry masa flour ($D_{gw}=0.23$ mm and $S_{gw}=1.98$).

Colors of the Dry Corn Masa Flours

The total color difference expressed as E value ($(E=L^2+a^2+b^2)^{1/2}$) showed that neither one of the two sources of dry corn masa flour was significantly affected ($P<1\%$) by oil extraction/reconstitution (Tables III and IV). Oil reconstitution did not affect 'L' values, but significantly increased 'a' and 'b' values of near fat-free commercial dry corn masa flour. In general, the near free-fat and reconstituted commercial flours (Table III) were as white as the untreated full-fat flour.

Lab-made dry corn masa flours (Table IV) were darker than commercial flours. Lab-made dry corn masa flours had more black specks that affected their appearance. The black particles were from the hilum left in the nixtamal after hand-washing. Near fat-free flour was more white than whole or oil-reconstituted flours. Oil reconstitution did not affect 'L' and 'a' values of lab-made dry masa flours, but 'b' significantly increased as oil reconstitution increased. Some of the colored compounds (primarily B-carotene) were extracted with the oil-hexane mixture, which resulted in whiter flour, as indicated by higher E values (Tables III and IV). Near fat-free dry corn masa flours were whiter than reconstituted flours, and exhibited the same trend as is reported for reconstituted fat-free wheat flours (MacRitchie 1983).

Dough Properties

Dough stickiness and tortillas rollability were not affected by oil extraction and/or oil reconstitution in either of the two sources of dry corn masa flour (Tables V and VI). Previous studies have shown that stickiness is primarily affected by cooking time of the corn and by moisture content of the dough (Bedolla et al 1983 and Ramirez, et al 1993). Rollabilities of tortillas made from commercial (Table V) or lab-made (Table VI) dry corn masa flours were not different at any of the oil levels.

Sensory Characteristics of Corn Tortillas

Oil significantly affected ($P < 5\%$) flavor scores of corn tortillas in both sources of dry corn masa flour (Tables VII and VIII). Flavor score of the near fat-free and 50% reconstitution treatments were statistically lower than the 100% reconstituted ($P \leq 5\%$). Flavor scores of both flour sources consistently trended towards higher scores as oil level increased. Thus, oil plays minor role to flavor of corn tortillas.

Oil significantly affected chewiness of corn tortillas (Tables VII and VIII). Tortillas made from reconstituted dry corn masa flours from both sources were significantly less chewy than the untreated full-fat dry corn masa flours (Tables VII and VIII). Chewiness decreased as oil level increased but did not equal the chewiness score of the full-fat corn tortillas. Both masa sources showed the same

trend in tortillas chewiness scores. Apparently, oil acted as a lubricating agent, which helped the starch-protein complex slide with less force during mastication causing low chewiness scores.

Oil significantly affected firmness of corn tortillas prepared from both sources of dry corn masa flours. Firmness scores of tortillas prepared from commercial (Table VII) and from lab-made (Table VIII) dry corn masa flours decreased as oil content increased. But, full reconstitution did not quite equal the score of the whole tortillas suggesting that how the oil is present in the masa dough is important to tortilla texture (Table VII). Oil forms "slip-planes" among the particles. Therefore, oil acted as a lubricating agent improving pliability and reducing firmness of the corn tortillas. Extraction of oil may improve shelf-life of dry corn masa flour but flavor, chewiness and firmness of corn tortillas are adversely affected.

CONCLUSIONS

Lab-made dry corn masa flour had similar chemical and physical properties as commercial masas; thus, our lab procedure for preparing dry corn masa flour was suitable for studying processing variables in the laboratory. Fat-free flours were whiter than full-fat dry corn masa flours. Oil did not affect dough stickiness or tortilla rollability. Flavor scores of corn tortillas increased with increased oil level. The presence of oil significantly improves chewiness and firmness of corn tortillas. Thus, oil is important to flavor, chewiness and firmness of tortillas, but not to physical properties of corn masa dough. Negative effects of oil extraction on flavor, chewiness and firmness of corn tortillas make it unfeasible to extend shelf-life of dry corn masa flour by extracting the oil.

LITERATURE CITED

- AMERICAN ASSOCIATION OF CEREAL CHEMISTS. 1983. Approved methods of the AACC, 8th ed. Method 30-20, approved 4-13-61, reviewed 10-27-82; Method 44-19, approved 4-13-61, reviewed 10-27-82; Method 08-01, approved 4-13-61, revised 10-28-81; Method 46-12, approved 10-8-76, revised 10-3-83; Method 02-52, approved 4-13-61, reviewed 10-27-82. The Association: St. Paul, MN.
- AMERICAN SOCIETY AGRICULTURAL ENGINEERS. 1990. Approved Methods of the ASAE. Method S319.2 approved 1990. The Association: St. Joseph, MI.
- BEDOLLA, S., PALACIOS, M. G., ROONEY, L. W., DIEHL, K. C., and KANAN, M. N. 1982. Cooking characteristics of sorghum and corn for tortilla preparation by several cooking methods. *Cereal Chem.* 60:263-268.
- GOMEZ, M. R., WANISKA, R. D., and ROONEY, L. W. 1991. Starch characterization of nixtamalized corn flour. *Cereal Chem.* 68:578-582.
- HUNTER, R. S. 1972. Visually perceived attributes of the appearance of materials and ASTM progress toward their measurement. In: *Sensory Evaluation of Appearance of Materials*. ASTM Special Technical Publication No. 545. American Society for Testing Materials: Philadelphia, PA.
- KHAN, M. N., DesROSIERS, M. C., ROONEY, L. W., MORGAN, R. O., and SWEAT, V. E. 1982. Corn tortillas evaluation of corn cooking procedures. *Cereal Chem.* 59:279-284.
-

- LINDSAY, R. C. 1985. Food Additives. Pages 629-688 in: Food Chemistry, 2nd ed. O. R. Fennema, ed. Marcel Dekker, Inc.: New York and Basel.
- MacRITCHIE, F. 1983. Role of lipids in baking. Pages 165-168 in: Lipids in Cereal Technology. Academic Press: New York and London.
- NAWAR, W. W. 1985. Lipids. Pages 139-244 in: Food Chemistry, 2nd ed. O. R. Fennema, ed. Marcel Dekker, Inc.: New York and Basel.
- RAMIREZ-WONG, B., SWEAT, V. E., TORRES, P. I., and ROONEY, L. W. 1993. Development of two instrumental methods for corn masa texture evaluation. Cereal Chem. 70:286-290.
- SAS INSTITUTE, INC. 1989. SAS/STAT User's Guide, version 6, 4th ed., Vol 1, and 2. The Institute: Cary, NC.
- STONE, H., and SIDEL, J. 1985. Introduction to sensory evaluation. Pages 66-94 in: Sensory Evaluation Practices. 2nd ed. Academic Press, Inc.: San Diego and New York.
- SEGUCHI, M. 1984. Oil-binding ability of heat-treated wheat starch. Cereal Chem. 61:248-250.
- SEGUCHI, M., and MATSUKI, J. 1977. Studies on pan-cakes baking. II. Effect of lipid on pan-cake qualities. Cereal Chem. 54:918-926.
- VIDAL-QUINTANAR, R. L., JOHNSON, L. A., LOVE, M. H., and LOVE, J. A. 1993. Lipid oxidation of dry corn masa flour. Cereal Chem. In press.
-

TABLE I
Proximate Compositions and pHs of Commercial Dry
Corn Masa Flour Treatments¹

Property	Untreated² Full-Fat	Near Fat-Free	50% Oil Recon.	100% Oil Recon.
Moisture (%)	6.7	6.7	6.6	6.7
Fat (%)	4.3	0.1	2.1	4.3
Protein (%) ³	8.7	8.4	8.3	8.7
Ash (%)	1.0	1.0	1.0	1.0
pH	8.0	7.4	7.3	7.0

¹ All values are means of three replicates and expressed on dry matter bases, except moisture.

² Commercial full-fat dry corn masa flour.

³ Total %N X 6.25.

TABLE II
Proximate Compositions and pHs of Lab-Made Dry Corn Masa Flours¹

Property	Untreated Full-Fat²	Near Fat-Free	50% Oil Recon.	100% Oil Recon.
Moisture (%)	1.3	4.6	5.6	5.3
Fat (%)	4.4	0.4	2.1	4.3
Protein (%) ³	8.4	8.5	8.4	8.4
Ash (%)	1.0	1.0	1.0	1.0
pH	8.7	8.7	8.8	8.8

¹ All values are means of three replicates and are expressed on dry matter bases, except moisture.

² Lab-made full-fat dry corn masa flour.

³ Total %N X 6.25.

TABLE III
Effect of Oil on Colors of Commercial Dry Corn Masa Flour¹

Sample	Hunter Color Values ²			E ³
	'L'	'a'	'b'	
Untreated full-fat ³	86.6 ^a	-0.09 ^c	11.6 ^b	87.4 ^a
Near fat-free	86.7 ^a	-0.11 ^b	12.0 ^b	87.5 ^a
50% oil recon.	86.8 ^a	-0.02 ^b	11.6 ^b	87.6 ^a
100% oil recon.	86.7 ^a	0.09 ^a	13.4 ^a	87.7 ^a
LSD ⁴	0.90	0.06	1.0	0.9
SEM ⁵	0.34	0.001	0.43	0.3

¹ Means of four replicates. ^{a,b,c} Means with the same superscript within a column are not significantly different at P<5%.

² Hunter color values: 'a'=redness/greenness(+=red, -=green); 'b' yellowness/blueness (+=yellow, -=blue); and E= $(L^2+a^2+b^2)^{1/2}$.

³ Commercial full-fat dry corn masa flour.

⁴ Least significant difference (P<5%).

⁵ Standard error of the mean ($DF_{error}=12$).

TABLE IV
Effect of Oil on Colors of Lab-Made Dry Corn Masa Flour¹

Sample	Hunter Color Values ²			E
	'L'	'a'	'b'	
Untreated Full-fat ³	80.6 ^a	0.31 ^a	16.4 ^a	82.2 ^b
Near fat-free	85.9 ^b	-0.22 ^b	13.6 ^b	86.3 ^a
50% oil recon.	85.6 ^b	-0.11 ^b	14.5 ^b	86.8 ^a
100% oil recon.	83.9 ^b	-0.02 ^b	15.4 ^a	85.3 ^a
LSD ⁴	1.5	0.21	0.80	0.9
SEM ⁵	1.0	0.02	0.08	0.3

¹ Means of four replicates. ^{a,b} Means with the same superscript within a column are not significantly different at P<5%.

² Hunter color values: 'a'=redness/greenness(+red, -=green); 'b' yellowness/blueness (+yellow, -=blue); and E= $(L^2+a^2+b^2)^{1/2}$.

³ Lab-made full-fat dry corn masa flour.

⁴ Least significant difference (P<5%).

⁵ Standard error of the mean (DF_{error}=12).

TABLE V
Effects of Oil on Dough Stickiness and Tortillas Rollability
of Commercial Dry Corn Masa Flour¹

Sample	Stickiness ² (%)	Rollability ³ (No. of cracks)
Untreated full-fat ⁴	31.5 ^a	1.2 ^a
Near fat-free	31.5 ^a	1.2 ^a
50% oil recon.	29.4 ^a	1.2 ^a
100% oil recon.	32.5 ^a	1.1 ^a
LSD ⁵	5.5	0.2
SEM ⁶	12.6	0.01

¹ Means of four replicates. ^a Means with the same superscript within a column are not significantly different at P<5%.

² Dough retained on the stickiness device bar.

³ Degree of tortillas cracks.

⁴ Commercial full-fat dry corn masa flour.

⁵ Least significant difference (P<5%).

⁶ Standard error of the mean ($DF_{error}=12$).

TABLE VI
Effects of Oil on Dough Stickiness and Tortillas Rollability of
Lab-Made Dry Corn Masa Flour¹

Sample	Stickiness ² (%)	Rollability ³ (No. of cracks)
Untreated full-fat ⁴	29.7 ^a	1.2 ^a
Near fat-free	30.5 ^a	1.3 ^a
50% oil recon.	31.9 ^a	1.3 ^a
100% oil recon.	32.3 ^a	1.2 ^a
LSD ⁵	5.0	0.14
SEM ⁶	10.4	0.02

¹ Means of four replicates. ^a Means with the same superscript within a column are not significantly different at P<5%.

² Dough retained on the stickiness device bar.

³ Degree of tortillas cracks.

⁴ Lab-made full-fat dry corn masa flour.

⁵ Least significant difference (P<5%).

⁶ Standard error of the mean (DF_{error}=12).

TABLE VII
Effects of Oil on Sensory Properties of Tortillas Prepared from
Commercial Dry Corn Masa Flour¹

Sample	Corn Flavor²	Chewiness²	Firmness²
Untreated ³	8.4 ^a	9.5 ^c	7.5 ^c
Near fat-free	6.8 ^b	11.6 ^a	12.0 ^a
50% oil recon.	7.0 ^b	10.5 ^b	10.9 ^b
100% oil recon.	7.3 ^{ab}	10.5 ^b	10.9 ^b
LSD ⁴	1.3	1.0	0.9
SEM ⁵	5.2	4.3	3.9

¹ Means of three replicates. ^{a,b,c} Means with the same superscript within a column are not significantly different at the 5% level.

² Subjective evaluation (12 panelists) in which flavor, chewiness, and firmness were evaluated using 15-cm line scale with anchored ends. Higher numbers indicate greater intensity.

³ Commercial dry corn masa flour.

⁴ Least significant difference ($P < 5\%$).

⁵ Standard error of the mean ($DF_{error} = 94$).

TABLE VIII
Effects of Oil on Sensory Properties of Tortillas Prepared from
Lab-Made Corn Masa Flour¹

Sample	Corn Flavor²	Chewiness²	Firmness²
Untreated full-fat ³	6.0 ^a	5.2 ^b	4.2 ^b
Near fat-free	4.5 ^b	7.1 ^a	6.4 ^a
50% oil recon.	5.3 ^{ab}	6.5 ^a	5.1 ^b
100% oil recon.	6.0 ^a	4.8 ^b	4.6 ^b
LSD ⁴	1.0	1.2	1.0
SEM ⁵	5.3	6.3	5.2

¹ Means of three replicates. ^{a,b} Means with the same superscript within a column are not significantly different at the 5% level.

² Subjective evaluation (12 panelists) in which flavor, chewiness, and firmness were evaluated using 15-cm line scale with anchored ends. Higher numbers indicate greater intensity.

³ Lab-made full-fat dry corn masa flour.

⁴ Least significant difference ($P < 5\%$).

⁵ Standard error of the mean ($DF_{\text{error}}=94$).

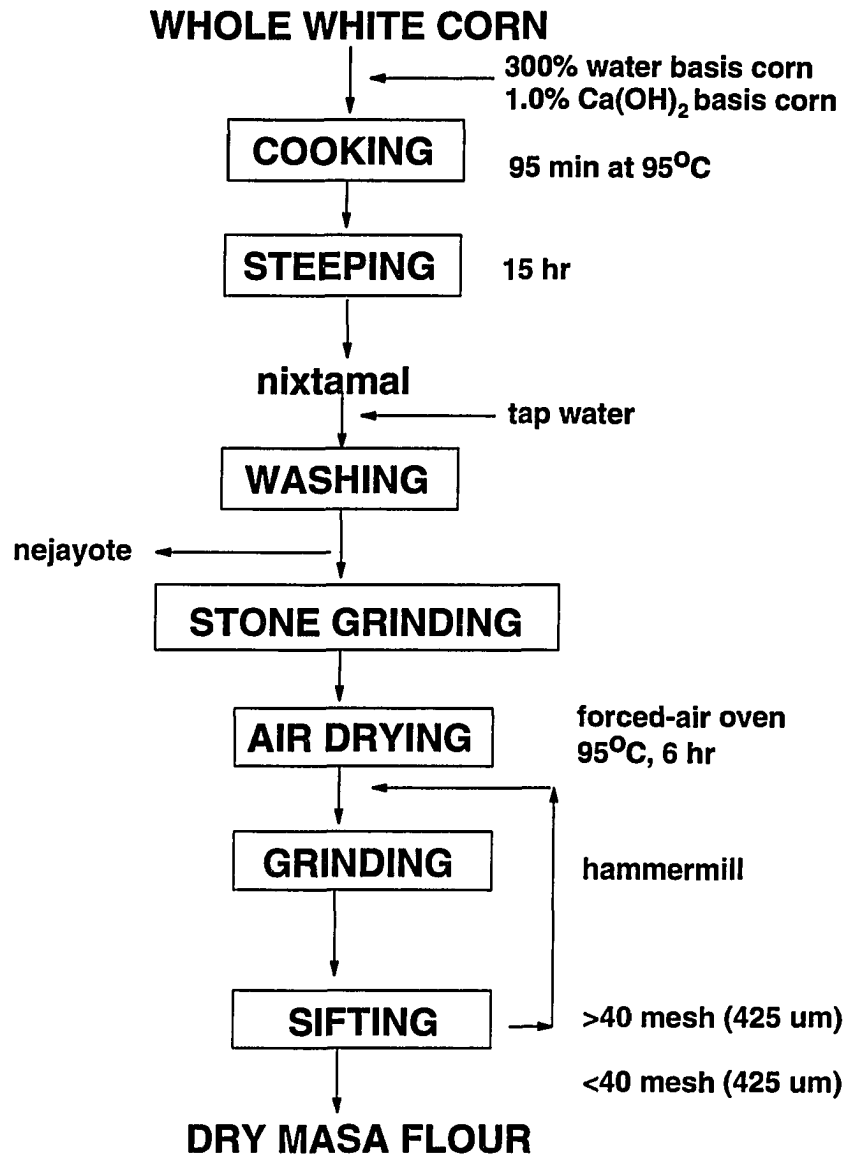


Fig. 1 Method for preparing lab-made dry corn masa flours.

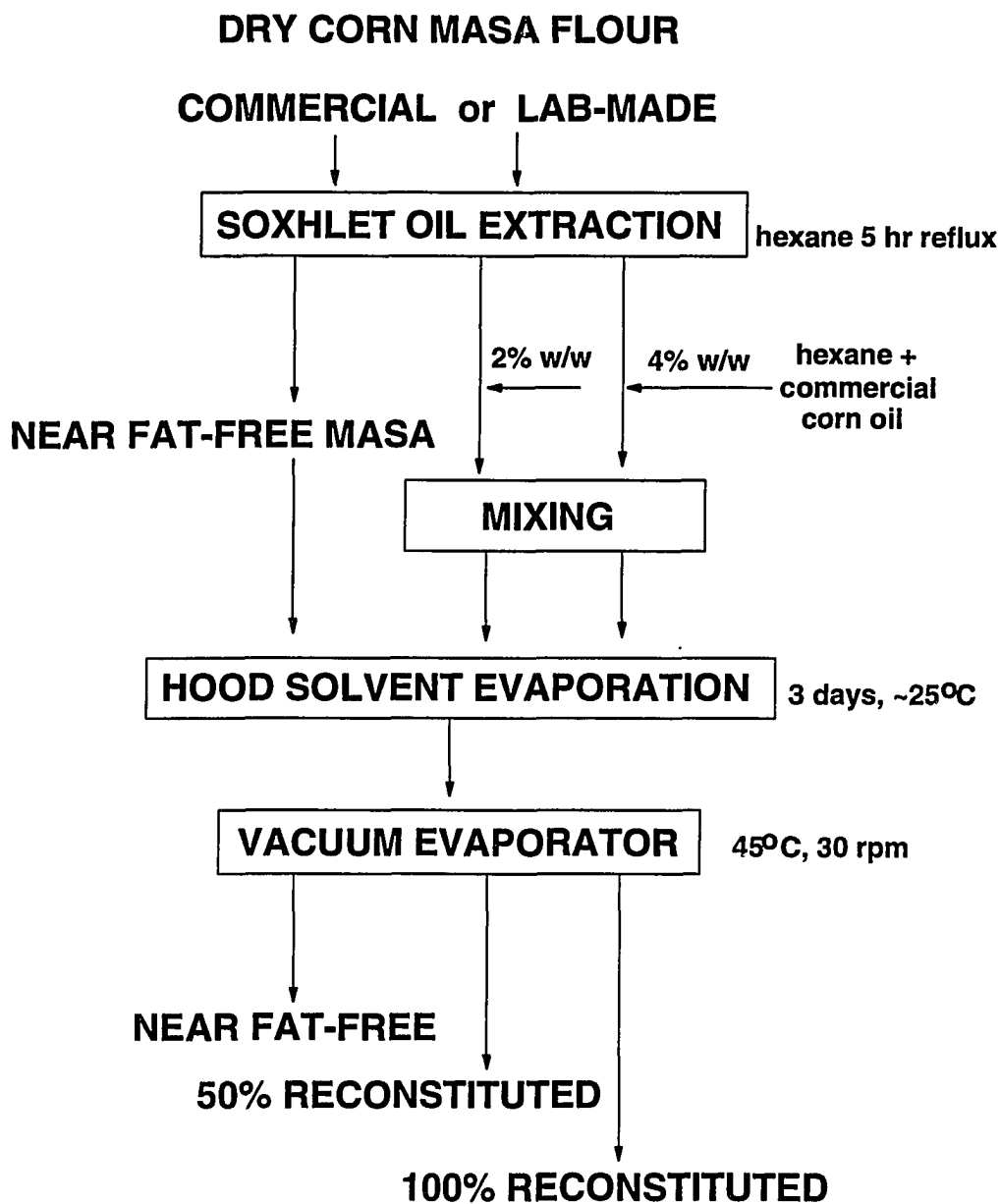


Fig. 2 Method for preparing near fat-free and reconstituted dry corn masa flours.

PAPER III

**ROLE OF CORN GERM ON PHYSICAL PROPERTIES OF DRY CORN MASA
FLOURS AND SENSORY CHARACTERISTICS OF CORN TORTILLAS**

**Role of Corn Germ on Physical Properties of Dry Corn Masa Flours
and Sensory Characteristics of Corn Tortillas¹**

**Reyna Luz Vidal-Quintanar², Lawrence A. Johnson^{2,3,4},
and Jane A. Love³**

¹ Journal Paper J- of the Iowa Agriculture and Home Economics Experiment Station, Ames, IA, 50011. Project 0178. Research supported by the Center for Crops Utilization Research and the Iowa Agriculture and Home Economics Experiment Station.

² Graduate Research Assistant and Professor-in-Charge, respectively, Center for Crops Utilization Research, Iowa State University, Ames, IA, 50011.

³ Professor and Associate Professor, respectively, Department of Food Science and Human Nutrition, Iowa State University, Ames, IA, 50011.

⁴ To whom correspondence should be addressed.

ABSTRACT

Laboratory-made (lab-made) whole and 50 and 100% degermed dry corn masa flours were prepared to determine the effects of germ constituents on physical properties and sensory characteristics of corn tortillas. Degermed dry corn masa flours were finer in particle size than whole dry corn masa flour. Whiteness of dry corn masa flours (E value) increased as degermination level increased. Removal of germs did not affect dough stickiness and tortillas rollability. Removal of germ did not significantly affect the typical flavor of corn tortillas. Sensory flavor scores consistently trended to be slightly higher values as more germs were present. Firmness and chewiness of corn tortillas decreased as the germ content were removed. The important role of germs on chewiness and firmness of corn tortillas made it unfeasible to extend shelf-life of dry corn masa flour by degerming the nixtamal.

INTRODUCTION

Commercial dry corn masa flours are often produced by the traditional batch method of cooking and steeping corn in lime water. After steeping the nixtamal, it is washed to remove loose pericarp fragments, tip caps, and excess of lime. Stone or hammer milling is the final step of processing wet corn masa. Wet masa is dried and marketed as rehydratable dry corn masa flour (Molina et al 1977).

Vidal-Quintanar et al (1993a) reported that autoxidation of oil causes off-flavor and off-odors of dry corn masa flours which reduce consumer acceptability of corn tortillas made from the flour. They also reported a 5.8-mo shelf-life when dry corn masa flour was stored at 25°C; but, shelf-life decreased to 2.5 mo when stored at 45°C.

In an attempt to extend the shelf-life of dry corn masa and determine the role of oil in corn tortillas, Vidal-Quintanar et al (1993b) extracted the oil in the flours with hexane and prepared tortillas. Oil did not significantly affect dough stickiness, tortillas rollability. Oil contributed only a small (but statistically significant) effect on the typical corn flavor of corn tortillas. However, oil played an important role in firmness and chewiness of corn tortillas. Firmness and chewiness of tortillas increased as oil level decreased (Vidal-Quintanar et al 1993b). We hypothesized that it may be possible to restore the original textural properties (firmness and chewiness) of defatted corn tortillas by also removing germ proteins along with the oil by degerminating of the nixtamal instead of merely extracting the oil. Germ proteins are highly water soluble

(Wilson 1987) and may have contributed to dough adhesiveness; thus, increasing firmness and chewiness when oil was removed but germ proteins remain.

The objective of the present paper was to determine the role of germ constituents on the physical properties of dry corn masa flours and sensory characteristics of corn tortillas in order to assess the feasibility of extending shelf-life of dry corn masa flour by degermination of nixtamal.

MATERIALS AND METHODS

Laboratory Process for Preparing Degermed Dry Corn Masa Flours

The procedure used for laboratory-made (lab-made) dry corn masa is shown in Fig. 1. White corn (Pioneer 3281, Pioneer Hi-bred International Inc., Johnston, IA) and 1% calcium hydroxide (Fisher Scientific, Fair Lawn, NJ) were mixed in a 1:3 corn:water ratio and then cooked by the traditional cooking method (95°C for 95 min). The nixtamal was soaked overnight, washed, and then divided into three portions. Portion one was kept as whole nixtamal, portion two had 50% of the germs removed by hand picking, and portion three was completely degermed. Milling and drying of each samples was done as previously described by Vidal-Quintanar et al (1993b).

Proximate Analyses and pH

Proximate analysis (moisture, oil, ash, and protein (%N x 6.25)) was determined for each treatment in triplicate by using AOAC methodology (AOAC 1988). pH was determined by dispersing 10 g of dry corn masa flour in 100 ml of distilled water.

Particle Size Distribution

Mean particle sizes (D_{gw}) and the geometrical standard deviation of the sample estimate by mass (S_{gw}) were determined using 100 g of dry corn masa flour (ASAE 1990). US standard sieves (50, 70, 100, and 200 mesh) with nominal sieve openings of 0.297, 0.210, 0.149, and 0.074 mm, respectively, were used in a Tyler Ro-tap sieve

shaker (T-673 model B, Combustion Engineering Inc., Mentor, OH). Mass of samples on each screen were determined after 10 min of shaking.

Color Evaluation

Color determinations were made on the surface of the flour under fluorescent lighting using the 10° standard observer. Samples were evaluated for Hunter color values where 'L' measured lightness, 'a' measured red (+) and green (-), and 'b' measured yellow (+) and blue (-) using a Hunterlab digital color difference meter (model JB 1201, Hunter Associates Laboratory, Reston, VA). Tiles 'L'=79.21, 'a'=-0.61, and 'b'=22.78 were used for standardization. The total color difference (E value) between the sample color and the corresponding difference tile was calculated as $E=(L^2+a^2+b^2)^{1/2}$ (Hunter 1972).

Dough Properties

Adhesiveness. Corn masa dough was made from 200-g of dry corn masa flour and sufficient water to reach 65 ± 1 % dough moisture as described by Ramirez et al (1993)

Tortilla Rollability. Rollability or dowel testing was performed as described by Vidal-Quintanar et al (1993b). Rollability testing involved a subjective evaluation of corn

tortilla texture (1 denoted no breakage, whereas 5 denoted high tendency for tortillas to break).

Sensory Characteristics

Selection and training of the sensory panel was done as described by Vidal-Quintanar et al (1993a). A group of three sets of freshly cooked tortillas samples (one set from each treatment) was presented to each panelist in random order under red light. Panelists evaluated each treatment by placing a vertical line across a 15-cm line scale anchored at the ends with terms which best described corn flavor, chewiness and firmness (Appendix A3) (from no corn flavor, not chewy, and tender/soft to intense corn flavor, chewy, and firm, respectively) (Stone and Sidel 1985). Sensory evaluations were carried out in an air-conditioned panel room equipped with individual booths.

Statistical Analysis

Analysis of variance (ANOVA) procedures (SAS Institute, Inc. 1988) were employed for data analysis. Data was analyzed as a randomized complete block design with three treatments (whole and 50% and 100% degermed nixtamal) of dry corn masa flours. Each treatment was replicated four times. When ANOVA revealed significant differences ($P < 1$ or 5%), means were separated using Fisher's Least Significant Difference procedure, LSD ($P < 5\%$).

RESULTS AND DISCUSSION

Properties of Degermed Dry Corn Masa Flours

The oil content of whole lab-made dry corn masa flour (5.5%) was slightly lower than that of the raw corn (Table I). Similar oil loss during tortilla preparation has been reported in other studies (Bressani et al 1958). The oil and protein contents of the nixtamal significantly decreased ($P < 5\%$) as more germs were removed (Table I). The corn germ is high in oil and protein (33% oil and 18.3% protein, Watson 1987).

Mean particle size of lab-made degermed dry corn masa flour (whole $D_{gw} = 0.19$ mm, $S_{gw} = 1.82$) was significantly smaller ($P < 5\%$) than whole dry corn masa flour ($D_{gw} = 0.23$ mm and $S_{gw} = 2.18$) (Table I). Degermination removed germ (which are characteristically large relative to endosperm particles) and reduced the resistance of the endosperm to the tearing forces during milling, producing flours with smaller mean particle sizes.

Colors of the Dry Masa Flours

Total color difference (E value) increased as more germs were removed (Table II). Whole dry corn masa flour was darker than 100% degermed flour. The 100% degermed dry corn masa flour had fewer black specks from hilum particles than whole flours, thereby increasing whiteness ('L' value). When oil and oil-soluble pigments (B-carotene) were removed, the intensity of yellow color ('b' value) decreased. Light-colored dry corn masa flours produced whiter tortillas (by visual determination).

Dough Properties

The extent of degermination did not affect ($P < 1\%$) dough stickiness and tortillas rollability of lab-made dry corn masa flours (Table III). Thus, germs did not affect dough stickiness and tortilla rollability of degermed dry corn masa flour. Vidal-Quintanar et al (1993) reported that oil alone did not affect dough stickiness or rollabilities of corn tortillas. Thus, germ proteins also play no role in dough stickiness or tortillas rollability.

Sensory Characteristics

Germs did not significantly affect ($P < 1\%$) the typical flavor of corn tortilla (Table IV). However, flavor scores of corn tortillas had slightly lower values as more germ was removed from the dry corn masa flour. A similar flavor trend was also observed in tortillas prepared from hexane-extracted dry corn masa flours (Vidal-Quintanar et al 1993b). Oil may make a minor contribution (albeit statistically insignificant) to the typical flavor of corn tortillas.

Removing the germ significantly increased chewiness of corn tortillas (Table IV). Whole and 50% degermed tortillas were significantly less chewy than 100% degermed tortillas. Apparently, oil acted as a lubricating agent reducing chewiness scores of corn tortillas.

Firmness of tortillas increased as the germs were removed (Table IV). Whole and 50% degermed tortillas were significantly more tender than 100% degermed

tortillas. Degermination of the nixtamal decreased oil content of the corn masa flour, which had a negative effect on corn tortilla firmness. Oil acts as a lubricating agent in masa flour and promotes tenderness/softness of corn tortillas.

Degermination of the nixtamal to produce dry corn masa flours gave physical properties and sensory characteristics similar to extracting the oil with hexane (Vidal-Quintanar et al 1993b). Degermination decreased oil content of the dry corn masa flour making the tortillas more chewy and less soft. Because of the same effect was observed in hexane-extracted dry corn masa flour (Vidal-Quintanar et al 1990b), germ proteins do not appear to play any role in chewiness, firmness, stickiness or rollability of corn tortillas. Degerming of the nixtamal cannot be used to extend the shelf-life of dry corn masa flour; because chewiness and firmness of corn tortillas will be adversely affected.

CONCLUSIONS

Colors of lab-made dry corn masa flours improved by the degerming the nixtamal. Degermination did not affect dough stickiness and tortillas rollability. Degermination of corn nixtamal did not significantly affect the typical flavor of corn tortillas, and degermed dry corn masa flours gave tortillas with acceptable flavor. However, flavor scores consistently trended downward (albeit statistically insignificant) as more germ was removed. Degermination levels decreased oil content of dry corn masa flour, adversely affecting chewiness and firmness of corn tortillas. The negative effects of degermination on chewiness and firmness of corn tortillas made it unfeasible to extend shelf-life of dry corn masa flour by degerming the nixtamal.

LITERATURE CITED

- AMERICAN ASSOCIATION OF CEREAL CHEMISTS. 1983. Approved methods of the AACC, 8th ed. Method 30-20, approved 4-13-61, reviewed 10-27-82; Method 44-19, approved 4-13-61, reviewed 10-27-82; Method 08-01, approved 4-13-61, reviewed 10-28-81; Method 46-12, approved 10-8-76, reviewed 10-3-83; Method 02-52, approved 4-13-61, reviewed 10-27-82. The Association: St. Paul, MN.
- AMERICAN SOCIETY AGRICULTURAL ENGINEERS. 1990. Approved Methods of the ASAE. Method S319.2 approved 1990. The Association: St. Joseph, MI.
- BEDOLLA, S., PALACIOS, M. G., ROONEY, L. W., DIEHL, K. C., and KHAN, M. N. 1983. Cooking characteristics of sorghum and corn for tortilla preparation by several cooking methods. *Cereal Chem.* 60:263-268.
- BRESSANI, R., and PAZ AND PAZ, R. 1958. Chemical changes in corn during preparation of tortillas. *J. Agri. Food Chem.* 6:770-773.
- GOMEZ, M. H., ROONEY, L. W., and WANISKA, R. D. 1987. Dry corn masa flours for tortilla and snack production. *Cereal Foods World* 32:372-377.
- HUNTER, R. S. 1972. Visually perceived attributes of the appearance of materials and ASTM progress toward their measurement. Pages 21-35 in: *Sensory Evaluation of Appearance of Materials*. ASTM special technical publication No. 545. American Society for Testing Materials: Philadelphia, PA.
- MOLINA, M. R., LETONA, M., and BRESSANI, R. 1977. Drum drying for the improved production of instant tortilla flour. *J. Food Sci.* 42:1432-1438.
-

PFLUGFELDER, R. L., ROONEY, L. W., and WANISKA, R. D. 1988. Fractionation and comparatione of commercial corn masa. *Cereal Chem.* 65:262-266.

RAMIREZ-WONG, B., SWEAT, V. E., TORRES, P. I., and ROONEY, L. W. 1993. Development of two instrumental methods for corn masa texture evaluation. *Cereal Chem.* 70:286-290.

SALDANA, G., and BROWN, H. E. 1984. Nutritional composition of corn and flour tortillas. *J. Food Sci.* 49:1202-1205.

SAS INSTITUTE, INC. 1989. *SAS/STAT User's Guide*, version 6, 4th ed., Vol 1, and 2. The Institute: Cary, NC.

SERNA-SALDIVAR, S. O., CANETT, R., VARGAS, J., GONZALES, M., BEDOLLA, S., and MEDINA, C. 1990. Effect of soybean and sesame addition on the nutritional value of maize and decorticated sorghum tortillas produced by extrusion cooking. *Cereal Chem.* 65:44-48.

STONE, H., and SIDEL, J. 1985. Introduction to sensory evaluation. Pages 66-94 in: *Sensory Evaluation Practices*. 2nd ed. Academic Press, Inc.: San Diego and New York.

VIDAL-QUINTANAR, R. L., JOHNSON, L. A., LOVE, M. H., and LOVE, J. A. 1993a. Lipid oxidation of dry corn masa flour. *Cereal Chem.* In press.

VIDAL-QUINTANAR, R. L., JOHNSON, L. A., and LOVE, J. A. 1993b. Role of oil on physical properties of dry corn masa flour and sensory characteristics of corn tortillas. *Cereal Chem.* In press.

WATSON, S. A. 1987. Structure and composition. Pages 53-60 in: Corn Chemistry and Technology. S. A. Watson and P. E. Ramstad eds. Am. Assoc. Cereal Chem.: St. Paul, MN.

WEBER, E. J. 1978. Corn lipids. Cereal Chem. 55:572-584.

WILSON, C. M. 1987. Proteins of the kernel. Pages 273-275 in: Corn Chemistry and Technology. S. A. Watson and P. E. Ramstad, eds. Am. Assoc. Cereal Chem.: St. Paul, MN.

TABLE I
Proximate Composition, pH, and Mean Particle Size
of Lab-Made Corn Masa Flours¹

Property	Lab-Made Dry Corn Masa Flour			
	Corn (Pioneer 3281)	Whole Nixtamal	50% Degermed	100% Degermed
Moisture (%)	10.9	1.3	1.2	1.0
Oil (%)	4.5	4.3	2.6	0.5
Protein (%) ²	8.9	8.7	8.4	7.5
Ash (%)	1.0	1.0	1.0	1.0
pH (LSD=0.27)	6.9 ^c	8.7 ^a	8.9 ^a	8.8 ^a
D _{gw} (mm) ³ (LSD=0.019)	-	0.23 ^a	0.19 ^b	0.19 ^b
S _{gw} ⁴ (LSD=0.25)	-	2.18 ^a	1.26 ^c	1.82 ^b

¹ All values are means of triplicates and are expressed on dry matter bases, except moisture. ^{a,b,c} Means with the same superscript within a column are not significantly different ($P < 5\%$, $DF_{\text{error}} = 12$).

² Total %N X 6.25.

³ Geometric mean diameter by mass of sample.

⁴ Geometric standard deviation of sample estimate by mass.

TABLE II
Effects of Germs on Colors of Lab-Made Dry Corn Masa Flours¹

Sample	Hunter Color Values ²			E
	'L'	'a'	'b'	
Whole nixtamal	80.6 ^b	0.31 ^a	16.4 ^a	82.2 ^b
50% degermed	82.2 ^a	0.11 ^b	16.4 ^a	83.8 ^a
100% degermed	83.4 ^a	0.14 ^b	15.5 ^b	84.8 ^a
LSD ³	1.2	0.18	0.3	1.5
SEM ⁴	0.6	0.02	0.05	0.7

¹ Means of four replicates. ^{a,b} Means with the same superscript within a column are not significantly different at P<5%.

² Hunter color values: 'L'=lightness (white/black); 'a'=redness/greenness (+=red, -=green); 'b' yellowness/blueness (+=yellow, -=blue); and $E=(L^2+a^2+b^2)^{1/2}$.

³ Least significant difference (P<5%).

⁴ Standard error of the mean ($DF_{error}=12$).

TABLE III
Effects of Germs on Dough Stickiness, and Tortilla Rollability
Prepared from Lab-Made Dry Corn Masa Flour¹

Sample	Stickiness ² (%)	Rollability ³ (No. of Cracks)
Whole nixtamal	29.7 ^a	1.20 ^a
50% degermed	29.5 ^a	1.25 ^a
100% degermed	30.9 ^a	1.25 ^a
LSD ⁴	5.6	0.14
SEM ⁵	13.5	0.01

¹ Means of four replicates. ^a Means with the same superscript within a column are not significantly different at $P < 5\%$.

² Dough retained on the stickiness device bar.

³ Degree of tortillas cracking.

⁴ Least significant difference ($P < 5\%$).

⁵ Standard error of the mean ($DF_{\text{error}} = 12$).

TABLE IV
Effects of Germs on Sensory Properties of Tortillas Prepared from
Lab-Made Dry Corn Masa Flours¹

Sample	Corn Flavor ²	Chewiness ²	Firmness ²
Whole nixtamal	6.0 ^a	5.2 ^b	4.2 ^b
50% degermed	5.4 ^a	5.6 ^{ab}	4.7 ^b
100% degermed	5.0 ^a	6.6 ^a	5.9 ^a
LSD ³	1.3	1.1	1.0
SEM ⁴	7.4	5.7	5.1

¹ Means of three replicates (each replicate was the mean of 12 responses).

^{a,b} Means with the same superscript within a column are not significantly different at P<5%.

² Flavor, chewiness, and firmness were evaluated using 15-cm line scale with anchored ends, higher numbers indicate greater intensity.

³ Least significant difference (P<5%).

⁴ Standard error of the mean ($DF_{\text{error}}=70$).

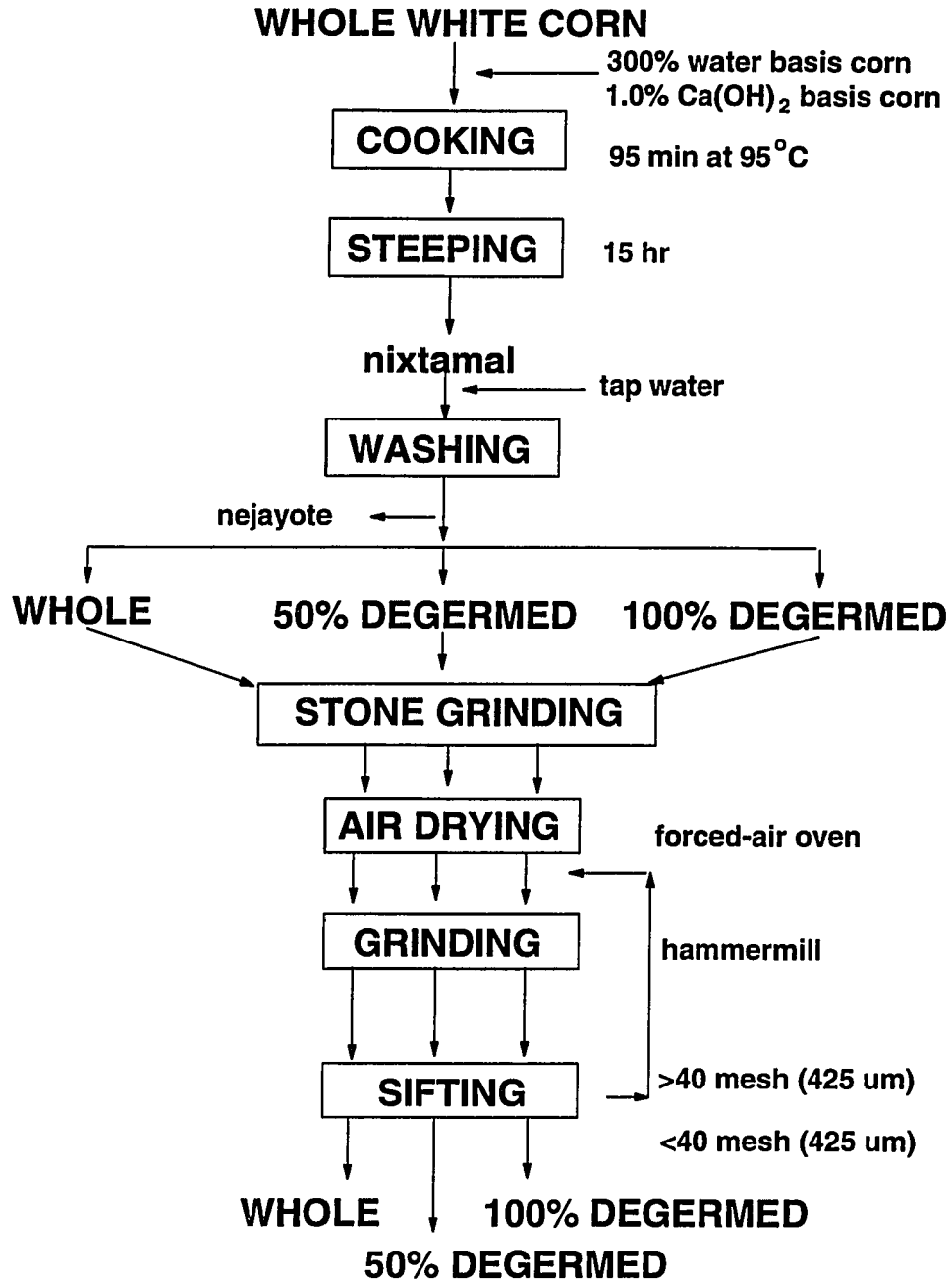


Fig. 1 Processes for preparing lab-made whole and degermed dry corn masa flours.

GENERAL SUMMARY

A retail marketplace survey showed that the oil of dry corn masa flour is typically quite oxidized. Rancidity of dry corn masa flours was greatest during summer and least during winter. Shelf-life of dry corn masa flour was 5.8 mo when stored at 25°C. A 20°C increase in temperature to 45°C reduced the shelf-life of dry corn masa flour to 2 mo. PV and CD were not good predictors or measures of rancidity in dry corn masa flours. Sensory evaluation of tortillas was a much more sensitive measure of rancidity than sensory analysis of flour. This study illustrated the need to develop new rapid methodology to measure lipid oxidation of dry corn masa flour.

The laboratory process for preparing dry corn masa appeared to be suitable for studying processing variables. Color and particle size of commercial dry corn masa and lab-made masa were not affected by oil extraction/reconstitution. Oil did not affect dough stickiness and tortillas rollability. Oil contribute a small, but statistically significant, effect to the typical corn tortilla flavor. Oil significantly improved tortilla chewiness and firmness. The negative effects of oil extraction on flavor, chewiness and texture of corn tortillas make it unfeasible to extend shelf-life of dry corn masa flour by extracting the oil.

Colors of lab-made dry corn masa flours were improved by degerming the dry corn masa flour. Degermination did not affect the dough stickiness and tortilla rollability of lab-made dry corn masa flours. Degermination did not affect the

typical flavor of corn tortillas. Degermination decreased the oil and protein contents of the dry corn masa flour and negatively affected chewiness and texture of corn tortillas. Oil improved firmness and reduced chewiness of corn tortillas. The positive effects of germ on texture and chewiness of corn tortillas make it unfeasible to extend shelf-life of dry corn masa by degerminating the nixtamal.

LITERATURE CITED

- ANONYMOUS. 1979. "La tecnología de la tortilla". Consejo Nacional de Ciencia y Tecnología de México.
- BAZUA, C., GUERRA, R., and STERNER, H. 1979. Extruded corn flour as an alternative to lime-heated corn flour for tortilla preparation. *J. Food Sci.* 44:940-941.
- BRESSANI, R., and PAZ AND PAZ, R. 1958. Chemical changes in corn during preparation of tortillas. *J. Agri. Food Chem.* 6:770-773.
- BRESSANI, R., GOMEZ-BRENES, R., and SCRIMSHAW, N. S. 1961. Effect of processing on distribution and In vitro availability of niacin of corn (*Zea mays*). *Food Technol.* 15:450-453.
- BRESSANI, R. 1990. Chemistry, technology, and nutritive value of maize tortillas. *Food Rev. Int.* 6:225-264.
- BEDOLLA, S., and ROONEY, L. W. 1982. Cooking Maize for masa production. *Cereal Foods World* 27:219-221.
- BEDOLLA, S., PALACIOS, M. G., ROONEY, L. W., DIEHL, K. C., and KHAN, M. N. 1983. Cooking characteristics of sorghum and corn for tortilla preparation by several cooking methods. *Cereal Chem.* 60:263-268.
- BEDOLLA, S., and ROONEY, L. W. 1984. Characteristics of U.S. and Mexican instant maize flours for tortilla and snack preparation. *Cereal Foods World.* 29:732-735.
-

- CRAVIOTO, R. O., MASSIEU, G. H., CRAVIOTO, O. Y., and FIGUEROA, M. 1952. Effect of untreated corn and mexican tortilla upon the growth of rats on a niacin-tryptophan deficient diet. *J. Nutr.* 48:453-459.
- CHU, N. T., and PELLETT, P. L. 1976. Effect of alkali treatment on the formation of lysinoalanine in corn. *J. Agric. Food Chem.* 24:1084-1085.
- DANIELS, J. R., and WHISTLER, R. L. 1985. Principal changes in starches during food processing. Pages 305-327 in: *Chemical Changes in Food during Processing*. J. W. Finley, ed. AVI Publishing Company, Inc: Westport, CT.
- DE GROOT, A. P., SLUMP, P., FERON, V. J., and VANBEEK, L. 1976. Effect of alkali-treated proteins: Feeding studies with free and protein-bound lysinoalanine in rats and other animals. *J. Nutr.* 106:1527-1538.
- ERIKSSON, C. E. 1987. Oxidation of lipids in food systems. Pages 207-233 in: *Autoxidation of Unsaturated Lipids*. H. W. S. Chang, ed. Academic Press: London, Orlando, and San Diego.
- GOMEZ, M. H., McDONOUGH, C. M., ROONEY, L. W., and WANISKA, R. D. 1989. Changes in corn and sorghum during nixtamalization and tortilla making. *J. Food Sci.* 54:330-336.
- GOMEZ, M. H., ROONEY, L. W., and WANISKA, R. D. 1987. Dry corn masa flours for tortilla and snack production. *Cereal Foods World* 32:372-377.
- HAMILTON, R. J. 1989. The chemistry of rancidity in foods. Pages 1-23 in: *Rancidity in Foods*. 2nd. ed. Elsevier Applied Science: London and New York.

- HARPER, A. E., PUNEKAB, B. D., and ELUEHJEM, C. A. 1958. Effect of alkali treatment on the availability of niacin and amino acid in maize. *J. Nutr.* 66:163-172.
- INGLET, G. E. 1972. Corn proteins related to grain processing and nutritional value of products. Pages 177-193 in: *Symposium: Seed Proteins*. G. E. Inglet, ed. AVI Publishing Co.: Westport, CT.
- KHAN, M. N., DesROSIERS, M. C., ROONEY, L. W., MORGAN, R. O., and SWEAT, V. E. 1982. Corn tortillas evaluation of corn cooking procedures. *Cereal Chem.* 59:279-284.
- KATZ, S. H., HEDIGER, M. L., and VALLEROY, L. A. 1974. Traditional maize processing techniques in the new world. *Science.* 184:765-773.
- JACKSON, D. S., CHOTO-OWEN, C., WANISKA, R. D., and ROONEY, L. W. 1988. Characterization of starch cooked in alkali by aqueous high-performance size exclusion chromatography. *Cereal Chem.* 65:493-496.
- JOHNSON, B., ROONEY, L. W., and KHAN, M. N. 1980. Tortilla-making characteristics of micronized sorghum and corn flours. *J. Food Sci.* 45:671-674.
- MAURON, J. 1977. General principles involved in measuring specific damage of food components during thermal processes. Pages 328-359 in: *Physical Chemical and Biological Changes in Food Caused by Thermal Processing*. T. Hyoyen and O. Kvaler, eds. Applied Science Publishers Limited: London, England.
- MARTINEZ-HERRERA, M. L., and LACHANCE, P. A. 1979. Corn (*Zea mayz*)
-

- kernel hardness as an index of the alkaline cooking time for tortilla preparation. *J. Food Sci.* 44:377-380.
- McDONOUGH, C. M., TELLEZ-GIRON, A., GOMEZ, M., and ROONEY, L. W. 1987. Effect of cooking time and alkali content on the structure of corn and sorghum nixtamal. *Cereal Food World.* 32:660.
- MOLINA, M. R., LETONA, M., and BRESSANI, R. 1977. Drum drying for the improved production of instant tortilla flour. *J. Food Sci.* 42:1432-1438.
- MORAD, M. M., ISKANDER, F. Y., ROONEY, L. W., and EARP, C. F. 1986. Physico-chemical properties of alkali-cooked corn using traditional and presoaking procedures. *Cereal Chem.* 63:255-259.
- ORTEGA, E. I., VILLEGAS, E., and VASAL, S. K. 1986. A comparative study of protein changes in normal and quality protein maize during tortilla making. *Cereal Chem.* 63:446-451.
- PAREDES-LOPEZ, O., and SAHAROPULOS-PAREDES, M. E. 1982. Scanning electron microscopy studies of limed corn kernels for tortilla making. *J. Food Tech.* 17:687-693.
- PAREDES-LOPEZ, O., and SAHAROPULOS-PAREDES, M. E. 1983. Maize a review of tortilla production technology. *Baker's Dig.* 57:16-25.
- PFLUGFELDER, R. L., ROONEY, L. W., and WANISKA, R. D. 1988. Dry matter losses in commercial corn masa production. *Cereal Chem.* 65:127-132.
- PFLUGFELDER, R. L., ROONEY, L. W., and WANISKA, R. D. 1988b.

- Fractionation and comparatione of commercial corn masa. *Cereal Chem.* 65:262-266.
- POKORNY, J. 1987. Major factors affecting the autoxidation of lipids. Pages 141-206 in: *Autoxidation of Unsaturated Lipids*. H. W. S. Chang, ed. Academic Press: London, Orlando, and San Diego.
- ROBLES, R. R., MURRAY, E. D., and PAREDES-LOPEZ, O. 1988. Physicochemical changes of maize starch during lime-heat treatment for tortilla making. *J. Food Sci. Tech.* 23:91-98.
- SANDERSON, J., WALL, J. S., DONALDSON, G. L., and CAVINS, J. F. 1978. Effect of Alkaline processing of corn on its amino acids. *Cereal Chem.* 55:204-213.
- SERNA-SALDIVAL, S. O., KNABE, D. A., ROONEY, L. W., and TANKSLEY, JR., T. D. 1987. Effects of lime cooking on energy and protein digestibilities of maize and sorghum. *Cereal Chem.* 64:247-252.
- SERNA-SALDIVAR, S. O., ALMEIDA-DOMINGUEZ, H. D., GOMEZ, M. H., BOCKHOLT, A. J., and ROONEY, L. W. 1990. A method to evaluate ease of pericarp removal on lime-cooked corn kernels. *Cereal Chem.* 67:334-337.
- SERNA-SALDIVAR, S. O., ROONEY, L. W., and GREENE, L. W. 1991. Effect of lime treatment on the availability of calcium in diets of tortilla and beans: rat growth and balance studies. *Cereal Chem.* 68:565-570.
- SMITH, P. S. 1982. Starch derivatives and their use in foods. Pages 237-269 in:

Food Carbohydrates. D. R. Linebeek, and G. E. Inglet, eds. AVI Publishing Co.: Westport, CT.

TREJO-GONZALES, A., FERIA-MORALES, A., and WILD-ALTAMIRANO, C. 1982.

The role of lime in the alkaline treatment of corn for tortilla preparation. *Advances in Chem. Series.* 198:245-263.

VIVAS, N. E., WANISKA, R. D., and ROONEY, L. W. 1987. Effect of tortilla production on proteins in sorghum and maize. *Cereal Chem.* 64:384-389.

WEBER, E. J. 1978. Corn lipids. *Cereal Chem.* 55:572-584.

WHISKTLER, R. L., and DANIEL, J. R. 1985. Carbohydrates. Pages 69-137 in: *Food Chemistry.* 2nd ed. O. R. Fennema, ed. Marcer Dekker, Inc: New York.

ACKNOWLEDGEMENTS

I would like to express my sincere appreciation to my major professor, Dr. Lawrence A. Johnson for his guidance and patience during my graduate career at Iowa State University. I also want to thank the other members of my graduate committee: Drs. Mark Love, Jane Love, Charles Hurburgh, and Linda Pollak for their extremely helpful suggestions.

I thank CONACyT and the Sonora State University for supporting me during the PhD program. I want to express my gratitude to my colleagues at the CIA (Centro de Investigaciones en Alimentos) of the Sonora State University for encouraging me to undertake graduate study.

I would like also to thank my groupmates, Chunyang Wang, Mark Reuber, Maide Osbay-Raeker, Katharine Miller, Shao-wen Wu, Feng Feng, Sonya Jennings, Steve Fox, Mila and Rock Evangelista for their friendship. I also want to thank my Latin American friends, Zoraida DeFreitas, Rodrigo and Mercedes Tarte, Lluvia and Antonio Regla and Raquel Leyva, for their support.

And finally, I want to thank my parents for their exceptional love and support and for raising me the way they did, and to my brother, sisters, nieces and nephews for the nice time we have shared together.

APPENDIX

**APPENDIX A1
Score Sheet (Category Scale) Used in Sensory Analysis
of Dry Corn Masa Flours**

Name _____ Date _____

OBJECTIVE: Evaluate the degree of lipid oxidation of dry corn masa flours.

INSTRUCTIONS: EXAMINE FOR RANCID AROMA. For each of the five samples, please do the following:
 a) SMELL the samples from left to right.
 b) PLACE an X on one line in each column that describes the rancid odor for the appropriate sample code.
 c) WRITE any comments at the bottom of the page.

EVALUATE FOR RANCID AROMA

SAMPLE CODES

	-----	-----	-----	-----	-----
1 not rancid	-----	-----	-----	-----	-----
2 slightly rancid	-----	-----	-----	-----	-----
3 rancid	-----	-----	-----	-----	-----
4 very rancid	-----	-----	-----	-----	-----
5 extremely rancid	-----	-----	-----	-----	-----

COMMENTS:

FOR SAMPLE # _____
 FOR SAMPLE # _____
 FOR SAMPLE # _____
 FOR SAMPLE # _____
 FOR SAMPLE # _____

**APPENDIX A2
Score Sheet (Category Scale) Used in Sensory
Analysis of Corn Tortillas**

Name _____ Date _____

OBJECTIVE: Evaluate the degree of lipid oxidation of tortillas made from dry corn masa.

INSTRUCTIONS: EXAMINE FOR RANCID AROMA. For each of the five samples, please do the following:
 a) **SMELL** and **TASTE** the samples from left to right.
 b) **PLACE** an **X** on one line in each column that describes the rancid odor for the appropriate sample code.
 c) **WRITE** any comments at the bottom of the page.

EVALUATE FOR RANCID AROMA

SAMPLE CODES

	-----	-----	-----	-----	-----
1 not rancid	-----	-----	-----	-----	-----
2 slightly rancid	-----	-----	-----	-----	-----
3 rancid	-----	-----	-----	-----	-----
4 very rancid	-----	-----	-----	-----	-----
5 extremely rancid	-----	-----	-----	-----	-----

COMMENTS:

FOR SAMPLE # _____
 FOR SAMPLE # _____
 FOR SAMPLE # _____
 FOR SAMPLE # _____
 FOR SAMPLE # _____

APPENDIX A3
Score Sheet (Scaling Scoring) Used in Sensory
Analysis of Corn Tortillas

NAME _____ DATE _____

OBJECTIVE: Evaluate the corn flavor, chewiness, and firmness of corn tortillas.

INSTRUCTIONS: make a vertical line on the horizontal line to indicate your rating of each attribute for each of the samples. Make any comments at the bottom.

=====

PLEASE TASTE SAMPLES IN THE FOLLOWING ORDER

SAMPLE SAMPLE SAMPLE SAMPLE SAMPLE

CORN TASTE

no corn

flavor

corn flavor

FIRMNESS

tender/soft

firm

CHEWINESS

not chewy

chewy

COMMENTS:

Sample #

Sample #

Sample #

Sample #

+++++

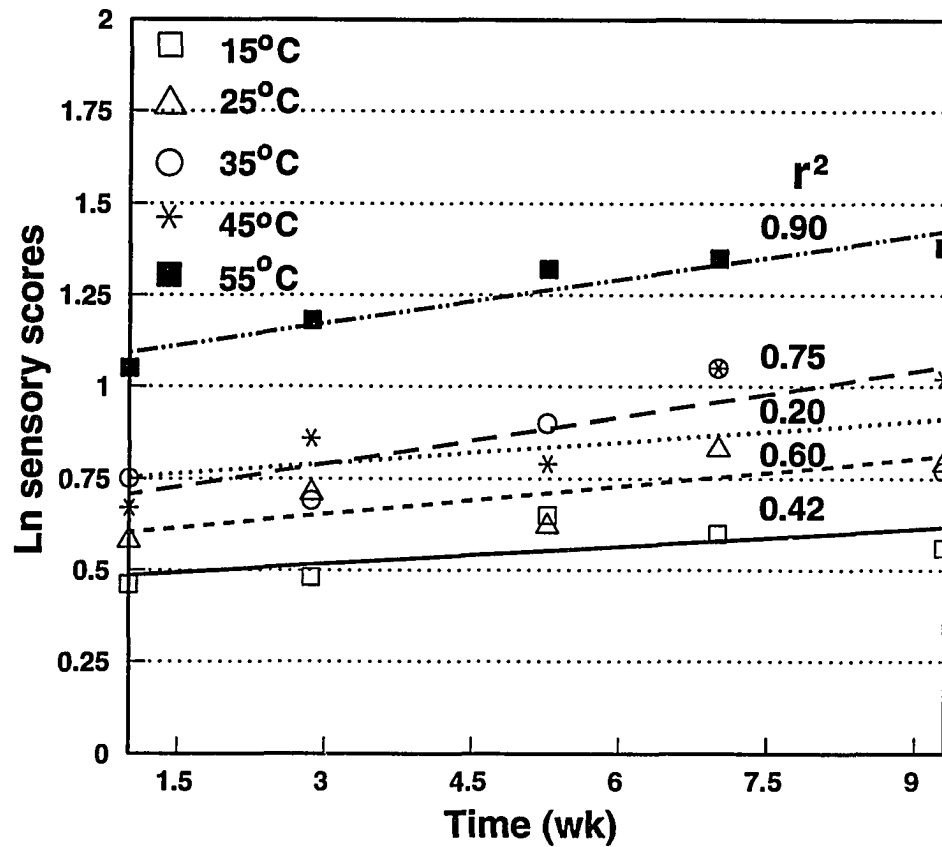


Fig. A1 First-order rate plots of lipid oxidation in Maseca corn masa flour as measured by sensory analyses.

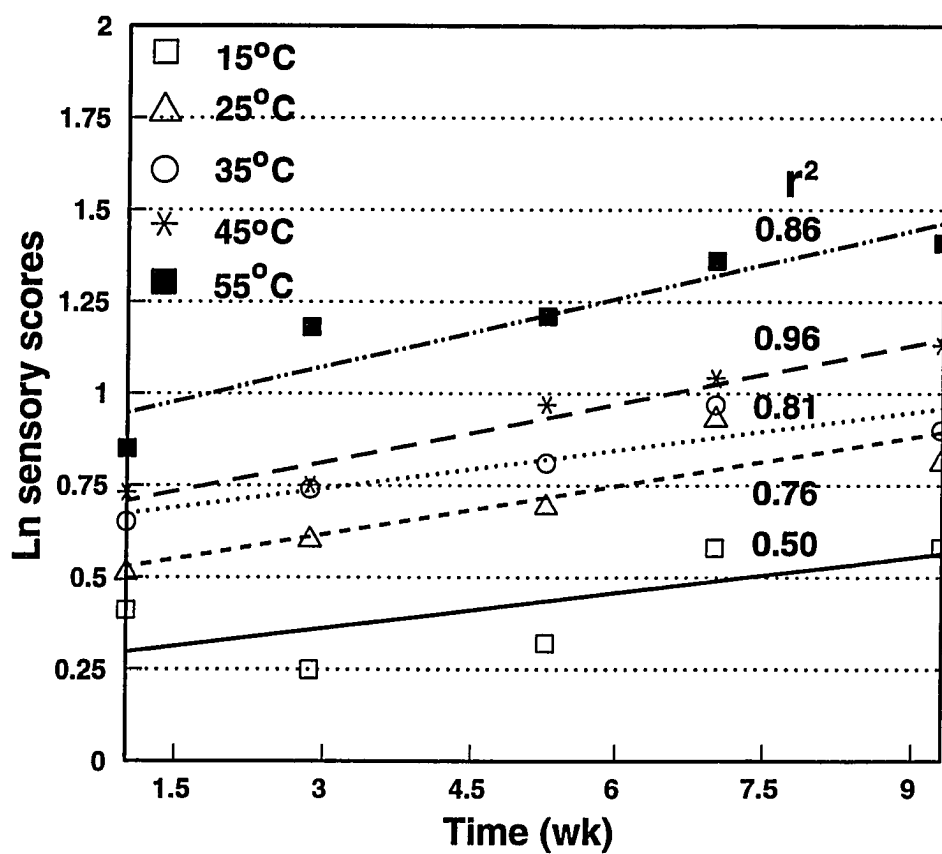


Fig. A2 First-order rate plots of lipid oxidation in tortillas prepared from Maseca dry corn masa flour as measured by sensory analyses.

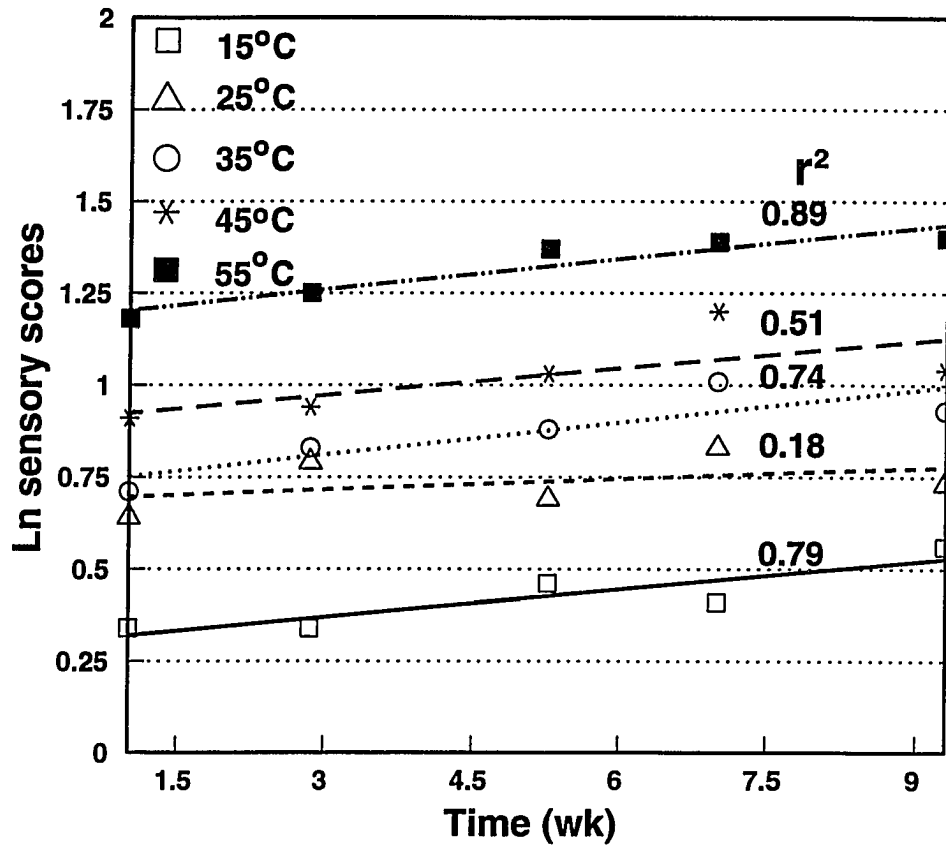


Fig. A3 First-order rate plots of lipid oxidation in Minsa dry corn masa flour as measured by sensory analyses.

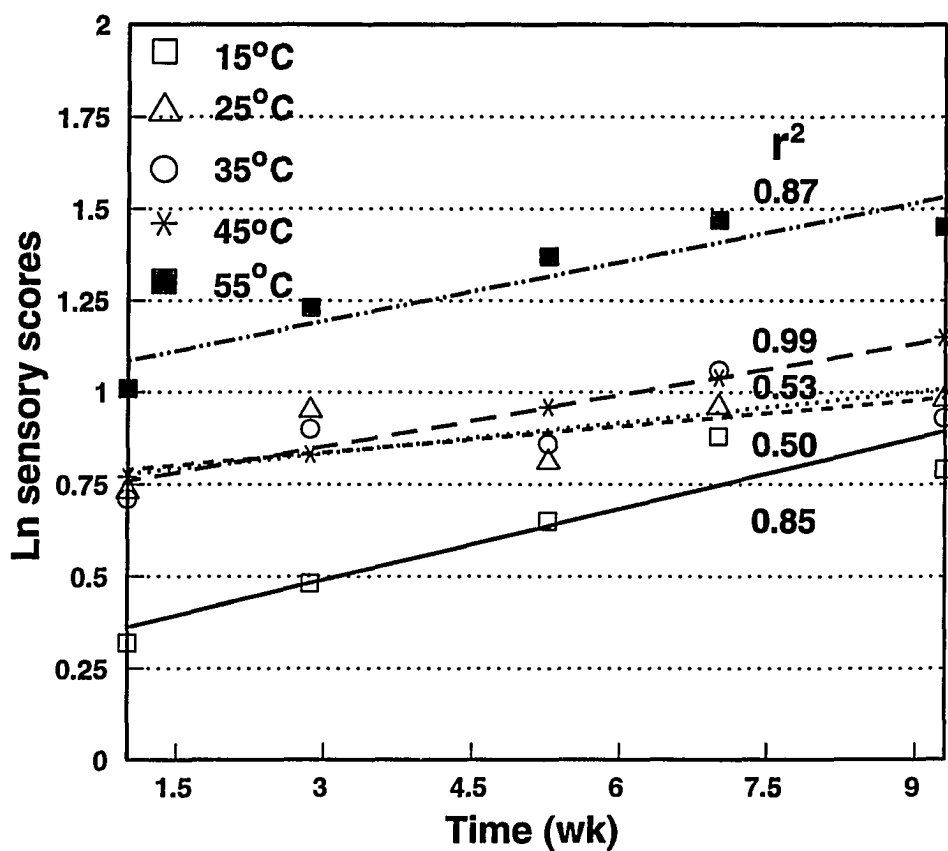


Fig. A4 First-order rate plots of lipid oxidation in tortillas prepared from Minsa dry corn masa flour as measured by sensory analyses.